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Evaluation of the 2014 EC measurement comparison on simulated airborne particulates: ^{137}Cs in air filters

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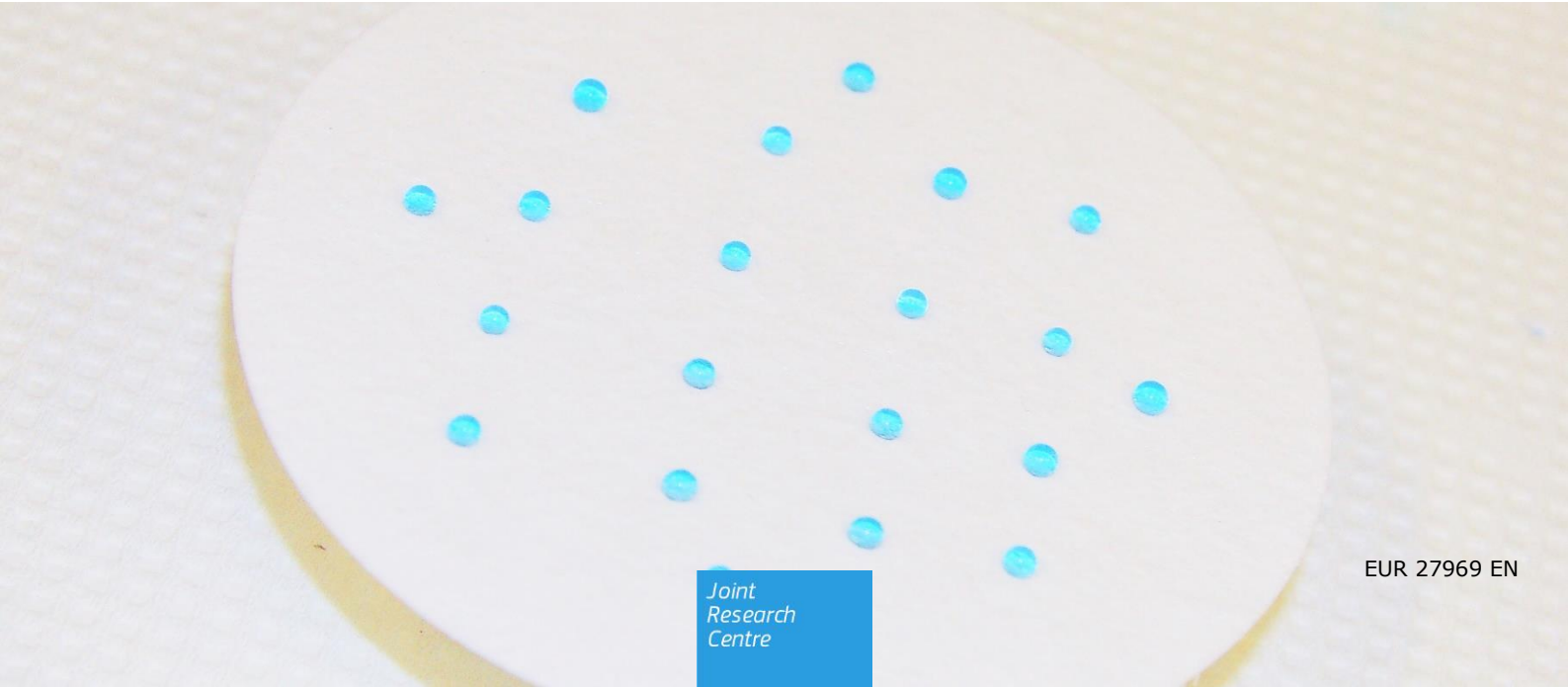

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Abstract

In 2014, the European Commission Directorate-General Joint Research Centre, Institute for Reference Materials and Measurements (JRC-IRMM) organized on request of the Directorate-General Energy an interlaboratory comparison (EC ILC) exercise on ^{137}Cs measurement in air filters; a similar exercise was organized in 2003.

This report describes the full life cycle of the above mentioned comparison among 76 European laboratories monitoring radioactivity in the environment. JRC-IRMM provided the comparison samples which were prepared individually for each laboratory using a gravimetrically diluted ^{137}Cs solution standardised by JRC-IRMM. The samples were made by gravimetrically dispensing the necessary activity amounts close to those the laboratories routinely measure. Reference values were fully documented and available to all participants and nominating national authorities after completion of the comparison. A robust evaluation of the individual performance using three different approaches, percentage difference ($D_{\%}$), E_n numbers and PomPlots, is presented. Finally, the performance of laboratories which have participated in both the 2003 and the 2014 ^{137}Cs measurement in air filters exercises are compared.

The majority of the laboratories reported reliable measurement results; only 5 out of 76 participants reported values with a percentage difference larger than ±33% of the reference value. Furthermore, 23 laboratories did not fulfil the criterion of the compatibility test based on E_n numbers. These results point out eventual problems with radioactivity measurements in the air filters and estimation of uncertainty which need to be addressed by the concerned laboratories.

1 Introduction

According to the Articles 35/36 of the Euratom Treaty (Euratom, 2012) and the Commission Recommendation 473/2000 (2000/473/Euratom, 2000) derived from the Euratom Treaty, the Member States (MS) of the European Union (EU) have the legal obligation to inform the European Commission (EC) on a regular basis on the radioactivity levels in their environment (drinking water, soil, air and mixed diet). In order to obtain more information on the MS's measurement methods and on the quality of their reported values for the radioactivity levels determined in their environment, the EC has established the International Comparison Scheme for Radioactivity Environmental Monitoring (ICS-REM) (Wätjen, 2008).

In the frame of ICS-REM, the Institute for Reference Materials and Measurements (JRC-IRMM), which is one of the seven institutes of the European Commission's Directorate-General Joint Research Centre (JRC), organises on request of the Directorate-General for Energy the EC Interlaboratory Comparisons (EC ILCs) since 2003. The aim of the EC ILCs is not only to evaluate the results submitted by the participants but also to provide help and advice to the participating MS laboratories via workshops and meetings on how to improve the measurements and methods applied by them. During the last decade, the test materials used in EC ILCs have included air filters (2003), soil samples (2010) and foodstuff samples, such as milk powder (2005), bilberry powder (2011), mineral water (2008) and drinking water (2012). The approach of JRC IRMM in organising the comparisons is sketched in *Figure 1*.

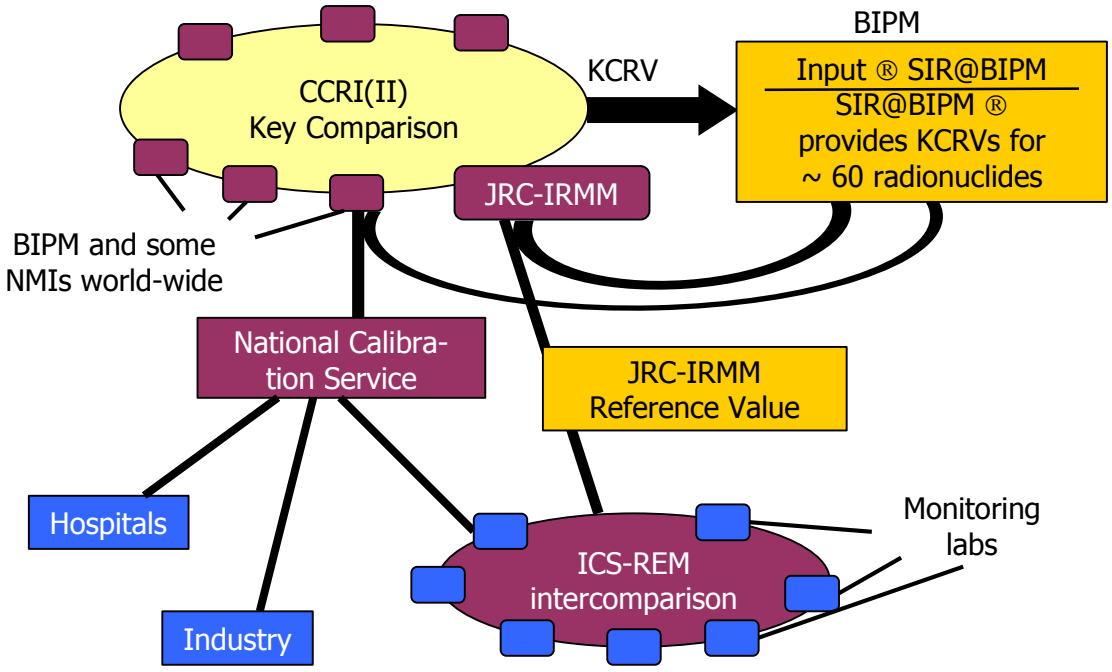

Fig. 1. Key comparisons of CCRI(II) and traceability of the reference values for samples provided by JRC-IRMM for the interlaboratory comparisons amongst monitoring laboratories (KCRV = Key Comparison Reference Value).

In 2014, following the agreement at the national experts meeting under Euratom Treaty Articles 35/36 on 25-26 October 2013, an EC ILC for the determination of ^{137}Cs in air filters was organised by JRC-IRMM, similar to the exercise organised in 2003 (Wätjen et al., 2007).

The Member States of the EU are obliged to report radiological monitoring data of airborne radioactivity to the EC according to the Council Decision 600/87

(87/600/Euratom, 1987) and the Commission Recommendation 473/2000, (2000/473/Euratom, 2000). These measurements are collected by the JRC Institute for Transuranium Elements (JRC-ITU) in the European Union Radiological Data Exchange Platform (EURDEP, 2015), which makes the non-validated radiological monitoring data available in nearly real-time for the countries reporting to the system. For the moment there are about 5000 operational stations to monitor the airborne radioactivity and dose rate within the European early warning network and EURDEP, but the number of stations with sampling equipment for radioactive particulates in air is significantly lower (~240) than those with instrumentation for dose rate measurements. Furthermore, the stations with sampling equipment use a wide variety of different methods, instruments and air filters.

The aim of the present EC ILC is to obtain an overview of the quality of the results reported, of the application of the measurement methods by the participating laboratories and of any changes that have occurred since the last similar exercise of 2003. The laboratories participating in this EC ILC are either laboratories from EU MS and nominated by their national representatives or other laboratories from geographical Europe that voluntarily report to the EURDEP system. For the current EC ILC, the comparison samples were provided by JRC-IRMM with fully documented reference values which values were available to all participants and nominating national authorities after the completion of the comparison.

This report describes in detail all phases of the ILC organised in 2014, the description of the intercomparison sample preparation at JRC-IRMM, the analytical methods used at the laboratories, the treatment of the data reported by the participants and, finally, the evaluation and comparison of the participants' results with the reference values. A robust evaluation of the performance of individual laboratories was performed using three different approaches: percentage difference ($D_{\%}$), E_n numbers (ISO, 2005a), and 'PomPlots' (Pommé, 2016).

2 The 2014 EC measurement comparison

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2.1 Description of the sample

Nature:	blank air filters provided by the participating laboratories spiked with a standardised ^{137}Cs solution at JRC-IRMM
Reference date:	1 January 2015 0:00 UTC
Recommended half-life of ^{137}Cs:	30.05 (8) a or 10975 (29) d (k=1) (DDEP, 2015)
Activity levels:	air filters spiked with activity levels similar to those which the laboratories routinely measure, and in every case, the activity level was designed to be above the reported detection limit (^{137}Cs activity ranging from 0.069 up to 2.310 Bq)
Shipping:	spiked air filters sealed in double plastic bags were sent via regular mail

2.2 Participating laboratories

The laboratories participating in this EC ILC are either laboratories from EU MS and nominated by their national representatives (national experts according to Euratom Treaty Article 35 and 36) or laboratories from geographical Europe voluntary reporting to the EURDEP system.

Fig. 2. Geographical location of the participants; EU laboratories are marked in red, non-EU laboratories are marked in green.

Authorities from 26 EU MS countries nominated 68 laboratories. Additionally, 8 laboratories from 6 non-EU countries participated as they voluntarily report to the EURDEP. In total, 76 participants from 32 European countries submitted their results (*Figure 2*). All laboratories who registered to the EC ILC reported their results. The list of all participating laboratories is given in *Annex 1*. Since the anonymity is a requirement in the EC ILC programmes, the identity of the laboratories is not shown in this compilation of the results. The laboratory numbers used throughout the data evaluation in this report are not related to the order of listing the participants in *Annex 1*.

2.3 Reporting of results

The unit of the results for the activity per filter as well as the associated combined uncertainty with coverage factor *k* had to be reported in Bq.

The reporting of the results together with a questionnaire was performed via an online reporting system, operated by JRC-IRMM. Participants were asked to answer all relevant questions regarding the used measurement procedure. Information given in

this questionnaire was essential for the evaluation of the EC ILC results. Moreover, it allowed to find the possible sources of difficulties and to get an overview of the methods used by the laboratories.

2.4 Timetable of ILC

11 September 2014:	invitation letter was sent to the national representatives
4 October 2014:	laboratories were nominated by the national representatives
5 November 2014:	nominated laboratories sent the blank air filters, together with information on their routine measurement conditions
December 2014:	spiked air filters were prepared
21-22 January 2015:	spiked air filters were sent to the participants via express mail (DHL) together with the information on the EC ILC
27 February 2015:	laboratories submitted their results and answered a second questionnaire with information on the air filter measurements
16 July 2015:	preliminary results were sent to participants

All registered communication with the participants related to this EC ILC can be found in *Annex 3*.

3 The Reference value

3.1 Standardisation of the spike solution

For this ILC exercise ^{137}Cs has been chosen as it is easy to measure and requires no specific corrections. The main difficulty lies in the low to very low activity levels (close to detection limit) and the possible deviation from the routine counting geometry and thus counting efficiency due to the imperfect spiking homogeneity of the filters with the radioactive solution. The standardisation of the ^{137}Cs solution used for the spiking of the air filters was performed by liquid scintillation counting (LSC). Gamma-ray spectrometry was used as a secondary method and as an additional link to the standard solution used for the ILC of 2003.

For the LSC, the efficiency tracing method developed by CIEMAT/NIST (Grau Malonda and Garcia-Toraño, 1982) was used. The principle of the CIEMAT/NIST efficiency tracing method is a combination of theoretical calculations of the radionuclide beta particle counting efficiency and an experimental determination of correction factors with the help of a tracer radionuclide, ^{3}H in this case. No impurities were detected in the solution by HPGe gamma-ray measurements checking for possible impurities and especially for ^{134}Cs.

All sources were prepared gravimetrically using a Mettler AX26 (Mettler-Toledo, Greifensee, Switzerland) mass comparator, calibrated using traceable weights. To prepare the LSC sources, aliquots of the radioactive solution were gravimetrically dispensed using the pycnometer method (Sibbens and Altzitzoglou, 2007; Campion, 1975) into 20-mL low-potassium glass LSC vials containing 15 mL of UltimaGold® (PerkinElmer, Boston, MA, USA) liquid scintillation (LS) cocktail, mixed with 1 mL of deionized water.

Three samples were prepared in this way from the original mother solution (A1), 11 from the first dilution (B1) and 8 from the second dilution (C1). The amount of radioactive solution in each sample ranged from 9 to 53 mg for A1, from 11 to 43 mg for B1 and from 23 to 50 mg for C1. All samples were measured 15 times (from 20 to 240 minutes each per run) using a Packard 3100 TR/AB (PerkinElmer, Boston, MA,

USA) liquid scintillation counter and 3 times (from 20 to 60 minutes each per run) using a Wallac Quantulus 1220 (PerkinElmer, Boston, MA, USA) LSC over a period of one month.

The standardisation methodology including the ^{137}Cs nuclear data, the liquid scintillation parameters and the specification of the counting device used for standardisation can be found in *Annex 3* of Wätjen et al. (2007). For the CIEMAT/NIST efficiency tracer method, the computer code CN2005 (Günther, 2002) was employed to calculate the radionuclide beta-particle counting efficiencies. As tracer, the IRMM tritiated water standard was used (Spernol and Denecke, 1964; Makepeace et al, 1998) and the maximum efficiency for ^{137}Cs was 1.15 for both LSC instruments used. The overall uncertainty of the method was determined to be 0.85% (k=1). In the calculations nuclear data as proposed by the Decay Data Evaluation Program (DDEP, 2015) were used, like the ^{137}Cs half-life of 30.05 (8) a or 10975 (29) d.

The activity concentration of the mother solution was found to be 3.60 (3) MBq g^{-1} on the reference date 1 January 2015 0:00 UTC. As usually, the numbers in parentheses are the numerical values of the combined standard uncertainties u_c expressed in the unit of the quoted result.

Traceability was guaranteed by using the exact same method as for participation at the ongoing comparison BIPM.RI(II)-K1.Cs-137 (Ratel et al., 2005), following the standardisation of ^{137}Cs for the EC ILC organised in 2003; the standardised solution of ^{137}Cs of the 2003 campaign was submitted together with the results of the described standardisation at JRC-IRMM as entry into the International Reference System (SIR) of the Bureau International des Poids et Mesures (BIPM) in order to establish traceability of the activity values of the spiking solutions and subsequently of the reference values of spiked activities on filters. The results of the measurements in the SIR at BIPM, summarised in *Annex 2*, are confirming traceability (to the key comparison reference value KCRV) within the combined relative measurement uncertainties of 0.8 %.

In addition, a gamma-ray spectrometric study was performed with the sources prepared gravimetrically using a Mettler AX26 mass comparator, calibrated using traceable weights. The radioactive solution was dispensed by means of a pycnometer onto a plastic foil supported by a 34-mm thin stainless steel annulus and covered by another plastic foil after drying completely. Two sources were prepared from the mother solution (A1) and three each from the dilutions B1 and C1.

Two high-purity germanium (HPGe) detector systems were used for the measurements, one with a 36% relative efficiency co-axial detector (Detector A) and the other with a 92% low-background co-axial detector (Detector B) (Canberra Industries, Inc., Meriden, CT, USA). The first detector was housed in a 10-cm thick Pb shield of circular cross-section, lined with 1 mm Cd and 1 mm Cu; the inner 2 cm of the Pb shield was made of high radiopurity Pb. The latter was housed in a 5-cm thick Pb shield of square cross-section. Both detectors were connected to commercial analogue electronics.

The spectra analysis was performed using the GammaVision-32 software program (ORTEC, Oak Ridge, TN, USA) and the data analysis was done with custom made spreadsheets. The sources were measured at two different distances from the detectors, a close one and a further one, against similar sources prepared during the 2003 EC ILC campaign and the discrepancy was determined to be less than 0.5%. The results are summarised in *Table A-7* and *Figure A-1*.

3.2 Dilutions

In order to approximate the activity level measured at each participating laboratory under routine conditions with the appropriate amount of spiked ^{137}Cs on the filters, four different diluted solutions (D1, D2, D3, D4) were prepared from the standardised mother solution (A1) via two intermediate dilutions (B1 and C1). The diluent was a

solution of 50 µg mL^{-1} Cs^{+} (as CsCl) in 0.1M HCl. In the final dilutions methylene blue, used to visualise the spikes on the filters, was gravimetrically added. *Table 1* lists the solutions and dilution factors including the addition of methylene blue. The numbers in parentheses are the numerical values of the combined standard uncertainties u_c expressed in the unit of the quoted result.

It should be noted that all dilutions were prepared gravimetrically for utmost traceability and small uncertainty on the dilution factors. In addition, quantitative sources were prepared from all dilutions for quality control by both liquid scintillation counting and gamma-ray spectrometry. The results of these measurements are presented in *Annex 9* and confirm the gravimetrical dilution factors.

Table 1. Dilutions and dilution factors with their combined standard uncertainties u_c (in parenthesis).

Solution	Code	Dilution factor D
Mother solution	A1	1
Dilution	B1	98.25 (4)
Dilution	C1	1303 (1) $x10^{1}$
Dilution	D1	3253 (2) $x10^{2}$
Dilution	D2	2817 (4) $x10^{3}$
Dilution	D3	4413 (6) $x10^{3}$
Dilution	D4	1372 (4) $x10^{4}$

In *Figure 3* the dilution step are shown schematically together with the approximate dilution factors and the activity concentration of each of the solutions prepared.

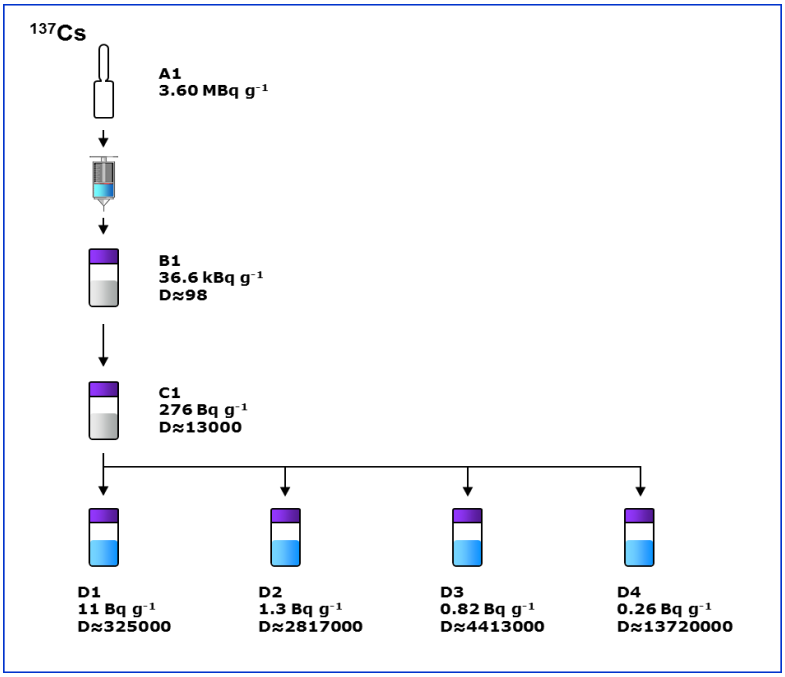

Fig. 3. Preparation scheme of the ^{137}Cs dilutions.

3.3 Spiking of the air filters

On the basis of the information provided by the participating laboratories in the relevant questionnaire, the ^{137}Cs activity per filter and the spiking pattern was determined for each filter individually. The amount of ^{137}Cs spiked onto each filter was chosen to resemble the activity routinely measured by the corresponding laboratory in a whole filter (or set of filters if it measures several at the same time). In cases where the laboratory declared to usually measure (sets of) filters with ^{137}Cs activities below detection limit, an activity higher than the declared detection limit was distributed.

The JRC-IRMM Radionuclide Metrology Sector prepared 76 air filters (*Annex 7*) by depositing on each of them gravimetrically an amount of ^{137}Cs from one of the standard solutions D1, D2, D3 and D4. Each participating laboratory in this ILC exercise had sent two blank filters of the type it is routinely using and after spiking one of them at IRMM with the ^{137}Cs solution, the filters were returned to the participants in order to be measured according to their routine procedure. The second blank filter was kept in reserve.

Since a uniform distribution of spikes was not feasible, depending on the filter size about 10 to 100 droplets were dispensed gravimetrically using a pycnometer, in a more or less symmetric pattern on the air filter. In any case, the spiked spots could easily be distinguished due to their methylene blue color, which allowed accounting for their discrete distribution when preparing the filters for measurement or when calculating counting efficiency corrections.

Where the filter was large enough, the filter was folded up in a way that the active part came into the centre of the pack such that any substance falling off the filter during transport by regular mail would still be caught in the surrounding filter and thus would not be lost. In the case of small filters this solution was not possible, therefore, the laboratories were asked to also measure the empty plastic bag in which the samples were shipped (or to measure the filter inside the bag) in order to verify that no losses from the filter had occurred.

Various spiked filters are shown in *Annex 8*. In many of the filters (e.g. those made of polypropylene or glass fibres) spiking became difficult due to the hydrophobic nature of the filters. The drops of radioactive solution were not absorbed into the filter material, therefore, the spiking had to be followed by several hours of drying at room temperature.

3.4 Reference values

The reference activity values, i.e. the spiked activities on the filters were calculated using the activity concentration of the mother solution determined by primary standardisation and the gravimetrically determined dilution factors (*Table 1*) of the spiking standard solutions D1, D2, D3 and D3. The mass and activity of the dispensed solution on each filter was determined once per filter by weighing the pycnometer before and after depositing the total number of drops on the corresponding filter. *Table 3* shows the reference values A_0 for the deposited ^{137}Cs activity on each filter and its combined standard uncertainty u_c. The standard uncertainty of activity includes the uncertainty contributions from the primary standardisation, the dilutions and the weighings of the filter spiked aliquots. The lowest ^{137}Cs activity spiked on an air filter was 0.069 (1) Bq and the highest value was 2.31 (2) Bq. The reference date for reporting the activity (and likewise of the reference values) is 1 January 2015 0:00 UTC. The numbers in parenthesis are the expanded uncertainties U with coverage factor $k=1$.

3.5 Quality control measurements

For the purpose of quality control sources were prepared at each stage of the preparation of the dilutions and the spiking of the filters. In addition to 33 liquid scintillation sources, 8 additional filters (Whatman ø70 mm) and 18 point sources were produced. The radioactive solutions were dispensed in the same way with a pycnometer into the LSC vials (20 mL low-potassium content glass vials) containing 15 mL Ultima Gold® cocktail with 1 mL H_2O added. *Table A-6* in *Annex 7* lists in sequence all filter samples prepared for the participating laboratories as well as all control samples. It shows clearly, how well the quality control samples interleave the rest of the samples prepared.

The 8 spiked control filters and the 18 point sources were measured by gamma-ray spectrometry at JRC-IRMM, using the two HPGe detectors mentioned in *Section 3.1*. Some of the sources were measured by both detectors. The point sources were measured at a close distance (2 mm) from the detector window and the counting efficiency calibration was performed with similar standard point sources. The filters were measured in their protective plastic bags and placed directly on the detector window. The acquisition time ranged from 1 to 7 days. The counting efficiency was obtained by measuring point sources; no attempt was made for geometry corrections, as the results were treated in a relative way, just to confirm the integrity of the dilutions.

The relative deviation of the measured activity from the reference activity, calculated from the mass of the spiked solution and its activity concentration and expressed in percent, is given for the point sources in *Table A-7* and shown in *Figures A-1* and *A-2* as the ratio of the measured to the reference activity. For the quality control air filters the percent deviations are listed in *Table A-10* and they are depicted in *Figure A-5*. The uncertainty is the expanded uncertainty combining both the uncertainty on the measured and reference values. All results are calculated for the reference date of 1 January 2015 0:00 UTC.

The LSC samples were measured using the Packard 3100 TR/AB LSC (from 20 and up to 240 minutes each and repeated for 8 times) and the Wallac Quantulus 1220 LSC (from 20 and up to 300 minutes each and repeated 3 times). The counting efficiency was calculated in the same way as for the standardization described in *Section 3.1*.

Again, the relative deviation in percent of the measured activity from the reference activity is given for the LSC sources in *Tables A-8* and *A-9* and shown in *Figures A-3* and *A-4* as the ratio of the measured to the reference activity.

The results from the gamma-ray spectrometry measurements are within 2 % from the reference values. For the LSC results, the measured activities of the solutions A1, B1 and C1 are within 0.6% for the reference values, while those of the lower activity D1, D2, D3, D4 solutions are within 10%. It is worth mentioning that LSC is not a suitable method for measuring low-activity sources of ^{137}Cs, because of the rather high background. Furthermore, sources with numbers 19 to 22 were unstable already just after their production and thus gave lower activity results. In conclusion, the measurement results confirm the activity concentrations determined from the gravimetric dilution and spiking procedure. One can safely conclude that the activity spiking procedure was applied successfully.

3.6 Influence of inhomogeneous spiked activity distribution on the counting efficiency

The activity distribution collected by air aspiration on air filters is presumably uniform and assumed mostly homogeneous, although exceptions may exist. The spiked air filters prepared for the present ILC campaign have been prepared by discretely depositing aliquots of a standard radioactive solution on the filters. Therefore, the activity distribution on the spiked filters is discrete and thus inhomogeneous. The

more drops deposited, the closer to a homogeneous distribution. This inhomogeneity is expected to affect the counting efficiency, but there are more parameters influencing the counting efficiency, which we will make an attempt to quantify.

One other parameter is the size of the deposited drops. Although the deposited solution mass, and thus activity, per drop is rather constant, depending on the quality and material composition of each filter the drop spreads to a smaller or larger area.

A third parameter for the determination of the spiked activity on the air filters is the counting geometry. For the smaller filters the usual geometry is placing the filter in front of the detector, either on the detector window or at a certain distance from the detector window. For larger filters the usual way is to fold in a suitable manner the filter and then place it in front of the detector. Unless the folding brings for some specific reason most of the active spots closer or further from the detector, it is expected that folding the filters distributes uniformly the active spots and consequently improves the homogeneity of the activity distribution.

A fourth parameter could be the size (active diameter) of the counting detector, giving an advantage to larger detectors, as in that case, because of the subtended solid angle, the active area of the filter is better covered by the detector.

In order to study the influence of those parameters on the counting efficiency for the measurement of the spiked air filters, a number of Monte Carlo simulations have been performed using the GEOLEP proprietary computer code (Solé, 1990; Lépy et al., 2010).

The counting efficiency has been calculated for three different HPGe detectors (of different Ge crystal size) and for 3 different filter sizes spiked in different patterns and with different (9 or 16) number and size (0.3, 0.7 and 1.0 mm in diameter) of active spots. The size of the detectors modelled was ø58.5 x 53.5 mm (Detector A), ø77 x 78 mm (Detector B) and ø80 x 30 mm (Detector C). The dimensions, matrix composition and density of the air filters, as well as the dimensions of the detectors have been used as model inputs to the Monte Carlo code. The simulations assumed that the gamma-ray emissions were isotropic and uncorrelated. The uncertainty of the Monte Carlo simulations was in all cases better than 1%.

The filters in *Figure 4* depict the different cases which have been modelled and *Tables 2A* and *2B* give more details, as well as the results of the Monte Carlo simulations. The last column in the tables gives the percent deviation of the calculated counting efficiency for the spiked air filters from that of an air filter of the same size but with a homogeneous activity distribution.

From *Tables 2A* and *2B* and the simulated counting efficiency results it is shown that for active filter areas smaller than or equal to the detector active areas, the discrepancies of the counting efficiency between discrete and homogeneous activity distribution is not large and in the studied cases, below 5%.

However, the worst case scenario was that of Detector C and the larger filter size, which resulted in an overestimation of the activity by 35.6%, most probably because the active spots are placed closer to the center of the detector, whereas on a homogeneous exposed filter the active area on the filter extends further from the center of the detector.

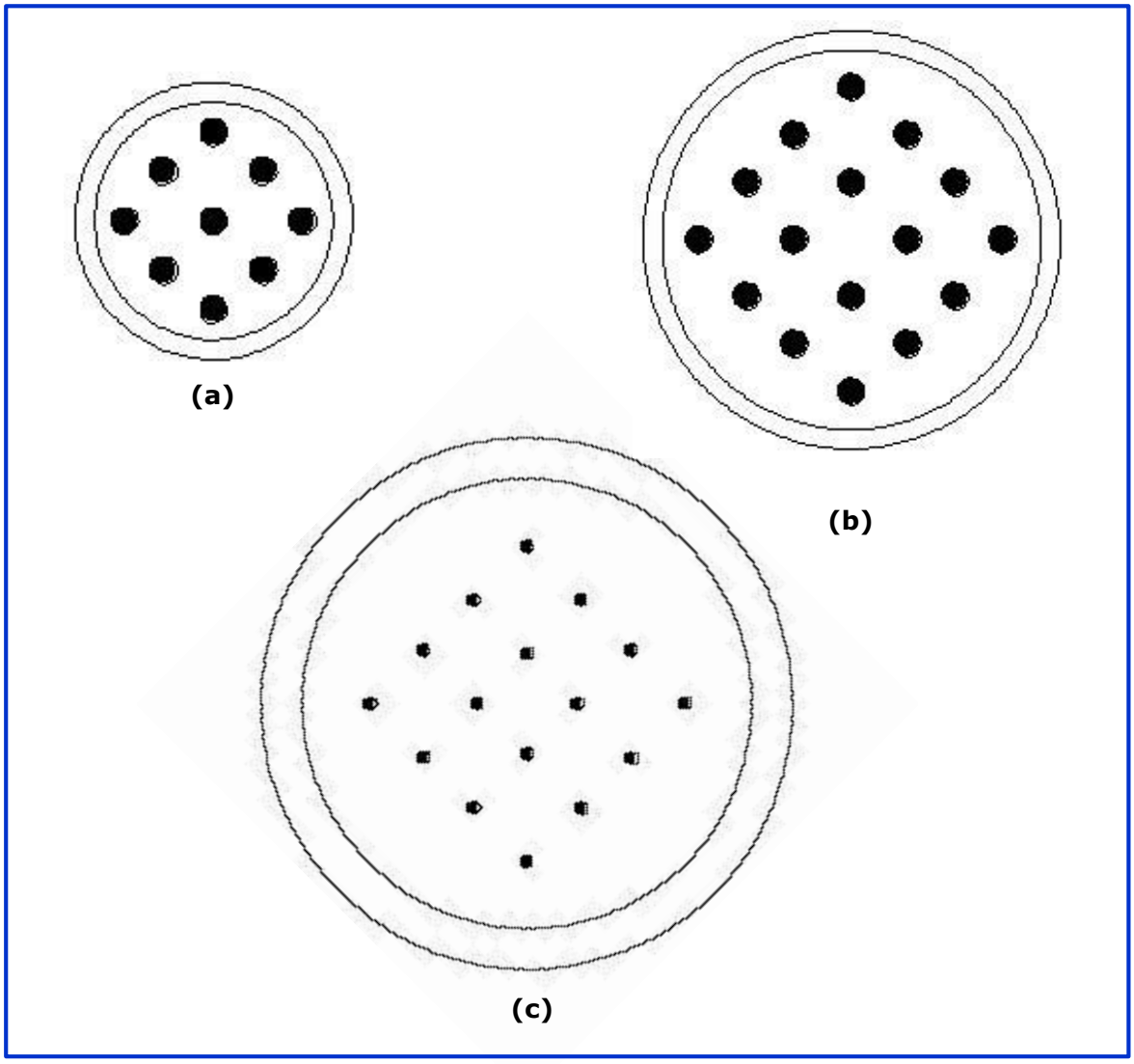

Fig. 4. Different cases of spiked air filters, which have been modelled for Monte Carlo simulations to calculate counting efficiencies with some typical detectors.
a) ø70-mm filter with 9 active spots
b) ø110-mm filter with 16 active spots and
c) ø130-mm filter with 16 active spots

Table 2A. Deviation of the efficiency of a spiked filter from that of a filter with homogeneous distribution of the activity as calculated using the Monte Carlo code GEOLEP (see text). Calculations are performed for two different detectors (A and B), two different sizes of the homogeneous active area (ø50 and 60 mm)

Detector and Ge crystal size (dia.xheight in mm)	Measure-ment geometry	Filter diameter (mm)	Homoge-neous active area diameter (mm)	Number of active spots on the spiked filter	Diameter of active spots on the spiked filter (mm)	Efficiency deviation from the homogeneous distribution (%)
Detector A ø58.5 x 53.5	on detector window	70	50	9	3	-4.27
					7	-4.50
					10	-5.09
			60	9	3	6.62
					7	6.38
					10	5.71
	at 10 mm	70	50	9	3	-2.92
					7	-3.16
					10	-3.63
			60	9	3	4.67
					7	4.42
					10	3.91
Detector B ø77 x 78	on detector window	70	50	9	3	-2.54
					7	-2.67
					10	-2.98
			60	9	3	4.01
					7	3.88
					10	3.54
	at 10 mm	70	50	9	3	-1.96
					7	-2.13
					10	-2.25
			60	9	3	2.86
					7	2.68
					10	2.56

and three different diameters of the 9 active spots (ø3, 7 and 10 mm).

Table 2B. Deviation of the efficiency of a spiked filter from that of a filter with homogeneous distribution of the activity as calculated using the Monte Carlo code GEOLEP (see text). Calculations are performed for two different detectors (A and B), two different sizes of the homogeneous active area (ø90 and 110 mm) and three different diameters of the 16 active spots (ø3, 7 and 10 mm). A third detector (C) was introduced as well and the efficiency for filters with homogeneous active area of ø110 mm was simulated.

Detector and Ge crystal size (dia.xheight in mm)	Measure-ment geometry	Filter diameter (mm)	Homoge-neous active area diameter (mm)	Number of active spots on the spiked filter	Diameter of active spots on the spiked filter (mm)	Efficiency deviation from the homogeneous distribution (%)
Detector A ø58.5 x 53.5	on detector window	110	90	16	3	-5.63
					7	-5.83
					10	-5.82
			100	16	3	4.24
					7	4.02
					10	4.03
	at 10 mm	110	90	16	3	-4.53
					7	-4.61
					10	-4.52
			100	16	3	3.94
					7	3.86
					10	3.96
Detector B ø77 x 78	on detector window	110	90	16	3	-5.55
					7	-5.66
					10	-5.55
			100	16	3	4.94
					7	4.82
					10	4.95
	at 10 mm	110	90	16	3	-3.82
					7	-3.89
					10	-3.82
			100	16	3	4.08
					7	4.01
					10	4.08
Detector C ø80 x 30	on detector window	130	110	16	15	35.60

4 Questionnaire on sampling method, air filter used and measurement conditions

In the preparative phase of the exercise the participants were asked to fill in a questionnaire (*Annex 3*) in order to receive air filters spiked with activity levels similar to those which the laboratories routinely measure and to allow them to measure following their usual procedure and measurement geometry. The questionnaire focused on two main fields: General information and Measurement details. As well as requesting general data about the laboratory, the questionnaire required information on the sampling method, the air filter used and the measurement conditions. The evaluation of the answers provided by the participants to the questionnaire is described in this chapter and in *Annex 4*.

The first part of the questionnaire collected the general information about the organisations and laboratories. The questions were grouped into four major topics: 1) contact details, 2) identity of laboratory, 3) accreditation and 4) previous participation in EC ILCs. On the basis of the answers to the question "What is the type of your laboratory?" it could be concluded that almost all participating laboratories monitor radioactivity in the environment routinely. Regarding accreditation, two thirds of the laboratories are accredited primarily for gamma-ray spectrometry measurements and one third of the laboratories are authorised by the government or responsible body for radioactivity measurements in the environment. On the basis of the responses received from participants, it was not possible to determine whether all of the accredited laboratories were, in fact, formally accredited to ISO 17025 (ISO, 2005b) by a government-appointed accreditation body. Furthermore, most of the participants have already had experience participating in EC ILCs and only 15 laboratories participated for the first time at this type of ILC. Thirty laboratories had participated in the previous air filter exercise in 2003.

The second part of the questionnaire contained the questions related to the technical part of the measurements of ^{137}Cs in air filter. Information on the air filter type used, the air sampling methods and the radioactivity measurement was gathered. As expected, air filters of various types and sizes are employed by the participating laboratories (*Annex 6*). The most popular air filter materials were glass fibre, nitrocellulose and polypropylene. According to size, they could be placed in three groups. On the basis of the second part of the questionnaire, it could be concluded that all of the laboratories determine the radionuclides in air filters routinely but there are no harmonised protocols. No correlation was found between the type and size of air filters, the sampling period, the total volume of air sampled per filter and the sampling frequency, except the fact that if the air filter size is larger, then the total volume of air sampled per filter is usually higher as well. The sampling frequency can be on a daily, weekly, monthly or annual basis or only occasionally.

Finally, in order to decide on the spiking of the individual filters, information on the minimum detectable activity (*MDA*), the typical activity level of ^{137}Cs measured per filter and any particular wish for a given pattern or transport were collected in the second part of the questionnaire. The ^{137}Cs activity per filter (or bunch of filters) measured routinely by the participating laboratories varied from 0.001 to a few Bq with a median of 0.12 Bq but in most cases it is below their detection limit of 0.001 to 1 Bq, (median 0.05 Bq). Additionally, besides the ^{137}Cs radionuclide, several natural and artificial radionuclides are determined such as ^{7}Be, ^{131}I, ^{40}K, ^{210}Pb, and ^{134}Cs.

5 Questionnaire on analytical and measurement procedures

The descriptions of the analytical and measurement procedures applied by the participants were collected by means of a questionnaire together with the reporting of the results (*Annex 10*). This questionnaire was divided into three parts: 1) sample treatment, 2) equipment used and 3) measurement and data evaluation including uncertainty budget. The evaluation of the answers provided by the participants to the questionnaire is described in this chapter and in *Annex 11*.

Most of the laboratories reported that they followed their routine procedures for the determination of ^{137}Cs in the spiked air filter. Some laboratories in their routine procedure collect more samples (filters) and stack them together for measurement. However, 6 of those laboratories (Laboratory 2, 14, 16, 17, 18 and 22) measured the single spiked air filter alone for this EC ILC exercise. Seven laboratories (Laboratory 4, 5, 13, 58, 62, 66 and 67) had difficulties to fit the air filters together with the plastic bag into their routinely used geometry or the geometry of the spiked air filter was not the same as their routine samples. In the case where the laboratories did not measure the spiked air filter in the plastic bag, it was advised to measure the plastic bag separately to ensure that no activity from the filter was left in the bag. All laboratories that measured the plastic bag separately reported no detectable activity on the bag or activity below their *MDA* level. Six of the participating laboratories (Laboratory 23, 25, 43, 52, 72 and 75) mentioned that because of the spiking method, the surface distribution of activity was not as homogeneous as their routine samples which made the determination of the counting efficiency and therefore the simulation of the ^{137}Cs activity on the spiked air filters difficult. The filter preparation for the measurement in the participating laboratories varied from no particular preparation at all, to pressing/compressing the spiked air filters, folding, packing together with blank air filters or the combination of the previous steps. Only one laboratory reported burning/ashing (Laboratory 73) and another one milling (Laboratory 63).

The measurements were mostly performed with commercially available detector systems and software. Only four participants (Laboratory 29, 54, 62 and 64) used self-developed software for the peak area determination and data evaluation. For the determination of the efficiency of the detectors almost half of the laboratories used single or multiple-nuclide sources and standard solutions. Reference air filter from blank filter spiked with certified radioactive solution for calibration purposes were prepared by 22 laboratories. Software was used by 18 laboratories to determine the counting efficiency by computer simulation. One laboratory (Laboratory 73), probably erroneously, reported that no efficiency calibration was performed.

Regarding the measurement parameters, the measurement cycles of the spiked air filters varied between 1 and 8 cycles, but in most of the cases (52 laboratories) the sample as well as the background were measured in one cycle. For the determination of the *MDA*, the laboratories used commercially available software and/or different calculation methods based on the Currie method, ISO 11929/2010, Risø method, DIN 25482 or other methods.

6 Results

6.1 Reported results

All 76 participants registered to this EC ILC reported valid results. *Table 3* organises the results in ascending order of the spiked ^{137}Cs activities. *Figure 5* shows in graphic form the spiked (reference) and the measured ^{137}Cs activity in the filters. The laboratory numbers have no correlation with the laboratory codes used during the exercise.

Two laboratories reported after the submission was closed that they noticed a reporting mistake. In the case of Laboratory 19 the actual value is 0.070 (4) instead of the reported 0.07 (4) and in the case of Laboratory 34 the actual value is 0.205 (25) instead of the reported 2.05 (25). For Laboratory 34 this explains the large deviation from the reference value. These examples prove the importance of careful and attentive reporting. Laboratory 73 reported difficulties during the measurement which could be the reason of the large deviation from the reference value. Additionally, Laboratory 27 sent a note that the calibration curve had been rechecked and the actual activity should be 0.194 (67) Bq instead of the reported 0.252 (102), which new value is closer to the reference value of 0.163 (3) Bq. However, the originally reported values in all the above cases cannot be altered and were used further in the analysis.

Table 3. Reference activity, reported activity result by the participating laboratories and ratio of the reported to the reference activity.

Lab Nr	Spiked activity A_0 (Bq)	Uncer-tainty $u_c(A_0)$ (Bq) k=1	Reported activity A (Bq)	Reported uncertainty $u_c(A)$ (Bq)	Report-ed k factor	Uncertainty $u_c(A)$ (Bq) k=1	Relative unc. $u_c(A)/A$ (%) k=1	Ratio (A/A_0)	Standard uncer-tainty $u_c(A/A_0)$
1	0.069	0.001	0.0827	0.0495	2	0.0248	29.93	1.19	0.30
2	0.073	0.001	0.078812	0.016335	2	0.008168	10.3633	1.08	0.10
3	0.074	0.001	0.0978	0.0142	1	0.0142	14.52	1.32	0.15
4	0.074	0.001	0.066	0.005	1	0.005	7.6	0.89	0.08
5	0.075	0.001	0.07	0.04	1	0.04	57	0.94	0.57
6	0.075	0.001	0.077577	0.01887	2	0.00944	12.162	1.03	0.12
7	0.083	0.001	0.085	0.006	2	0.003	3.5	1.03	0.04
8	0.084	0.001	0.0868	0.0098	1	0.0098	11.29	1.03	0.11
9	0.085	0.001	0.114	0.012	1	0.012	10.5	1.35	0.11
10	0.098	0.001	0.104	0.017	2	0.009	8.2	1.06	0.08
11	0.107	0.001	0.214	0.038	2	0.019	8.9	2.00	0.09
12	0.118	0.001	0.151	0.029	2	0.015	9.6	1.28	0.10
13	0.119	0.001	0.11	0.008	2	0.004	3.6	0.92	0.04
14	0.121	0.001	0.125	0.01	1	0.01	8	1.03	0.08
15	0.122	0.001	0.134	0.036	2	0.018	13.4	1.10	0.13

Lab Nr	Spiked activity A_0 (Bq)	Uncer-tainty $u_c(A_0)$ (Bq) k=1	Reported activity A (Bq)	Reported uncertainty $u_c(A)$ (Bq)	Report-ed k factor	Uncertainty $u_c(A)$ (Bq) k=1	Relative unc. $u_c(A)/A$ (%) k=1	Ratio (A/A_0)	Standard uncer-tainty $u_c(A/A_0)$
16	0.123	0.001	0.128	0.009	1	0.009	7.0	1.04	0.07
17	0.128	0.001	0.124	0.014	1	0.014	11.3	0.97	0.11
18	0.130	0.001	0.099	0.007	1	0.007	7.1	0.76	0.07
19	0.132	0.001	0.12247	0.02175	1	0.02175	17.759	0.93	0.18
20	0.138	0.001	0.16	0.04	2	0.02	13	1.16	0.13
21	0.138	0.001	0.144	0.017	2	0.009	5.9	1.04	0.06
22	0.140	0.001	0.13	0.013	1	0.013	10.0	0.93	0.10
23	0.147	0.001	0.168	0.014	2	0.007	4.2	1.14	0.04
24	0.154	0.001	0.161	0.009	1	0.009	5.6	1.05	0.06
25	0.158	0.001	0.153	0.01	1	0.01	7	0.97	0.07
26	0.160	0.001	0.18	0.015	1	0.015	8.3	1.12	0.08
27	0.163	0.001	0.252	0.102	2	0.051	20.2	1.55	0.20
28	0.165	0.001	0.206	0.0187	1	0.0187	9.08	1.25	0.09
29	0.169	0.001	0.164	0.026	2	0.013	7.9	0.97	0.08
30	0.170	0.001	0.132	0.013	1	0.013	9.8	0.78	0.10
31	0.171	0.001	0.14	0.03	1	0.03	21	0.82	0.21
32	0.171	0.001	0.184	0.021	2	0.011	5.7	1.08	0.06
33	0.178	0.002	0.21	0.01	2	0.01	2	1.18	0.03
34	0.179	0.002	2.05	0.25	2	0.13	6	11.47	0.06
35	0.182	0.002	0.19	0.03	2	0.02	8	1.05	0.08
36	0.185	0.002	0.184	0.041	2	0.021	11.1	0.99	0.11
37	0.190	0.002	0.163	0.015	2	0.008	4.6	0.86	0.05
38	0.192	0.002	0.15	0.02	2	0.01	7	0.78	0.07
39	0.193	0.002	0.179	0.016	2	0.008	4.5	0.93	0.05
40	0.194	0.002	0.188	0.012	1	0.012	6.4	0.97	0.06
41	0.196	0.002	0.163	0.016	1	0.016	9.8	0.83	0.10
42	0.198	0.002	0.196	0.015	1	0.015	7.7	0.99	0.08
43	0.206	0.002	0.15	0.015	1	0.015	10.0	0.73	0.10
44	0.215	0.002	0.212	0.02	1	0.02	9	0.99	0.09
45	0.224	0.002	0.245	0.026	1.65	0.016	6.4	1.10	0.06
46	0.242	0.002	0.235	0.026	1	0.026	11.1	0.97	0.11

Lab Nr	Spiked activity A_0 (Bq)	Uncer-tainty $u_c(A_0)$ (Bq) k=1	Reported activity A (Bq)	Reported uncertainty $u_c(A)$ (Bq)	Report-ed k factor	Uncertainty $u_c(A)$ (Bq) k=1	Relative unc. $u_c(A)/A$ (%) k=1	Ratio (A/A_0)	Standard uncer-tainty $u_c(A/A_0)$
47	0.267	0.002	0.25	0.01	1	0.01	4	0.94	0.04
48	0.271	0.002	0.315	0.025	1	0.025	7.9	1.16	0.08
49	0.279	0.002	0.266	0.049	2	0.025	9.2	0.95	0.09
50	0.280	0.002	0.302	0.012	1	0.012	4.0	1.08	0.04
51	0.284	0.002	0.29	0.08	2	0.04	14	1.02	0.14
52	0.297	0.003	0.27	0.06	2	0.03	11	0.91	0.11
53	0.298	0.003	0.338	0.023	1.645	0.014	4.1	1.13	0.04
54	0.315	0.003	0.34	0.03	1	0.03	9	1.08	0.09
55	0.322	0.003	0.34	0.05	2	0.03	7	1.06	0.07
56	0.417	0.004	0.45	0.1	1.96	0.1	10	1.08	0.11
57	0.426	0.004	0.5	0.07	1	0.07	14	1.18	0.14
58	0.441	0.004	0.318	0.015	1	0.015	4.7	0.72	0.05
59	0.488	0.004	0.47	0.05	2	0.03	5	0.96	0.05
60	0.529	0.005	0.57	0.015	1	0.015	2.6	1.08	0.03
61	0.553	0.005	0.4723	0.0303	1	0.0303	6.42	0.85	0.06
62	0.597	0.005	0.654	0.039	1	0.039	6.0	1.09	0.06
63	0.599	0.005	0.53	0.06	2	0.03	6	0.88	0.06
64	0.603	0.005	0.6	0.06	2	0.03	5	1.00	0.05
65	0.617	0.005	0.65	0.07	2	0.04	5	1.05	0.05
66	0.619	0.005	0.565	0.029	2	0.015	2.6	0.91	0.03
67	0.664	0.006	0.571	0.035	1	0.035	6.1	0.86	0.06
68	0.674	0.006	0.7	0.04	1	0.04	6	1.04	0.06
69	0.677	0.006	0.493	0.037	1	0.037	7.5	0.73	0.08
70	0.839	0.007	0.803	0.0549	1	0.0549	6.84	0.96	0.07
71	1.119	0.010	1.16	0.13	2	0.07	6	1.04	0.06
72	1.206	0.010	1.09	0.07	1	0.07	6	0.90	0.06
73	1.323	0.011	0.099	0	1	0	0	0.07	0.01
74	1.411	0.012	1.7424	0.11	2	0.06	3	1.23	0.03
75	1.502	0.013	1.46	0.07	1	0.07	5	0.97	0.05
76	2.310	0.020	2.46	0.36	2	0.18	7	1.06	0.07

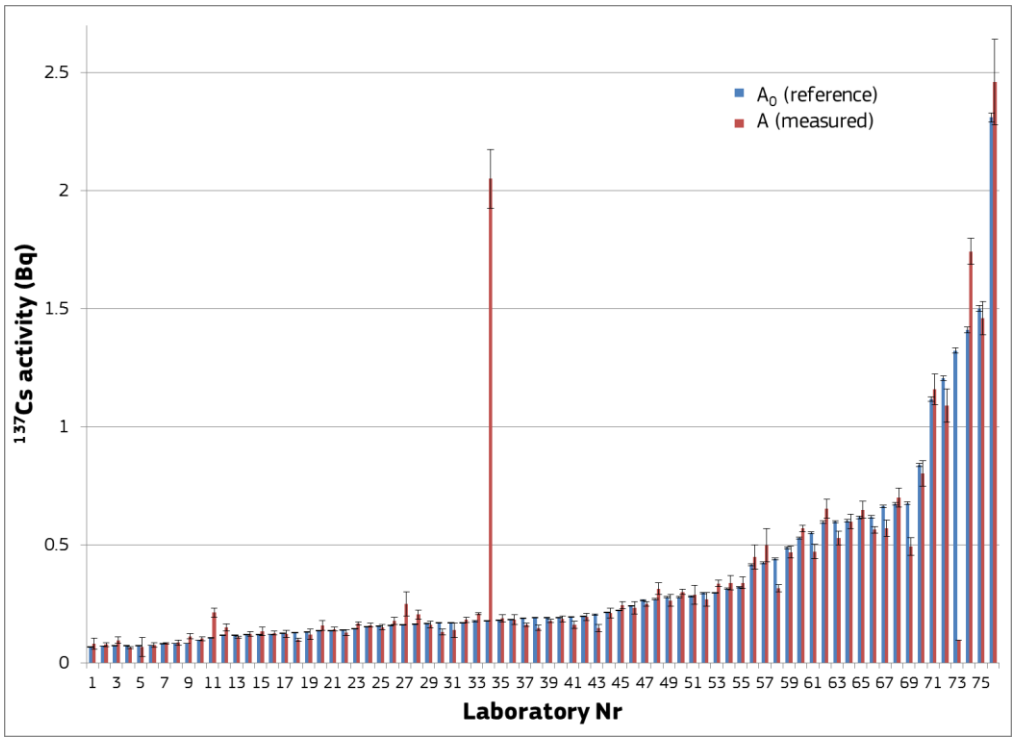

Fig. 5. ^{137}Cs activity spiked in the air filters: individual reference values A_0 (in blue) and activity results A (in red).

6.2 Uncertainty budgets

Participants were requested to provide in the corresponding table in the questionnaire the components of the uncertainty budget and the relative combined uncertainty (quadratic sum of the components). Although all of the participants submitted final results with the associated uncertainty, 11 of them completely omitted this part of the questionnaire and 14 laboratories provided incomplete uncertainty budget. The detailed uncertainty budgets submitted by the laboratories can be found in *Annex 12*.

The submitted uncertainty budgets were analysed on the basis of three components: 1) uncertainties reported with the results, 2) combined relative standard uncertainties reported in the questionnaire and 3) the combination (quadratic sum) of the individual, reported relative uncertainty components (*Table A-13*). Surprisingly, in many cases the simple conversion of the submitted uncertainties to relative standard uncertainties did not agree (inconsistent) with the values provided in the questionnaire. The cases where the effect of rounding could play a role were considered as consistent. The high number of discrepant uncertainty budgets most probably results from the fact that the determination of the uncertainties was not well treated by these laboratories or not enough attention was paid to these calculations (*Table 4*).

During the analysis of the uncertainty budgets we observed that Laboratory 19 submitted the uncertainty indicating a coverage factor k=1, but from the uncertainty budget it can be assumed that this value corresponds to k=2. In spite of this observation the reported uncertainties were used further in the analysis.

Laboratory 5 reported their result as 0.07 (4), instead of the actual value 0.070 (4), but no further information on the uncertainty budget was given which could have helped to realise the mistake at an earlier stage.

Table 4. Uncertainty budgets submitted by the participants.

Responses filled in the table requesting data on uncertainty budget and combined relative standard uncertainty in the questionnaire	Total number of laboratories	Consistent	Inconsistent
Both uncertainty budget and combined relative standard uncertainty were provided	51	41	10
Only uncertainty budget was provided	5	1	4
Only relative standard uncertainty was provided	5	3	2
Irrelevant values were provided	4	-	-
The table requesting information on uncertainty budget and relative standard uncertainty was not filled	11	-	-
Total number of laboratories	76 (100%)	46 (61%)	16 (21%)

7 Evaluation and comparison of the results

Initially, the results were tested for normality and presence of outliers. The outlying values were not discarded, but were included in further evaluations, unless it is declared differently. In the analysis the measurement uncertainty and that of the reference value were taken into account. Individual laboratory performance is expressed in terms of percentage difference ($D_{\%}$) and E_n number. The PomPlot method is used for producing a graphical overview of the results.

7.1 Identification of outliers and normal distribution test

In order to evaluate the performance of the laboratories, their measurement results need to be compared to the individual reference activity value, i.e. the spiked activity on each filter. The individual reported activities were normalised to their respective reference spiked activity and the measured (reported)–to-reference activity ratios, along with their combined standard uncertainties, u_c (k=1) are given in the last two columns of *Table 3* and plotted in *Figure 6*. u_c (k=1) was determined according to the following equation:

$$u_c(A/A_0) = \sqrt{\left[\frac{u_c(A_0)}{A_0}\right]^2 + \left[\frac{u_c(A)}{A}\right]^2} \quad (1)$$

where

A is the participant's reported activity result
$u_c(A)$ is the standard uncertainty of a participant's result (k=1)
A_0 is the assigned activity reference value

$u_c(A_0)$ is the standard uncertainty of the assigned reference value (k=1)
$u_c(A/A_0)$ is the combined standard uncertainty of the ratio A/A_0 (k=1)

Due to the relatively small uncertainty of the reference values, the uncertainty of the activity ratios measured versus spiked is dominated by the uncertainty of the measured values.

From *Figure 6* it can be seen that only five laboratories reported results which were more than 33% discrepant from the reference value. The limit of 33% is used for comparability with the 2003 exercise. The value was chosen as it was assumed that gamma-ray spectrometry – even with the low ^{137}Cs activity deposited by spiking on the filters using a technique which cannot provide as homogeneous distribution of the activity on the surface as the normal air sampling – can easily be performed within such large uncertainty. From the five outlier laboratories, one reported that they had problems with the measurement (Laboratory 73, -93% difference), Laboratory 27 rechecked the calibration curve, the new result would give a difference of 19% instead of 55% and last but not least another laboratory informed for a reporting mistake after submission of the results (Laboratory 34, 1047% was reported instead of the actual 15%). Although we know the reason of the very large deviation in the case of these laboratories, the originally reported values cannot be altered during the exercise and they are used further in the calculations.

No clear explanation of deviation could be found for the overestimation of Laboratories 9 (35%) and 11, (100 %) both of them followed their routine procedure for the determination of ^{137}Cs in the spiked air filters. The filter preparation for measurement by Laboratories 9 and 11 contained cutting. These two laboratories were the only to report cutting as part of the sample preparation. However, neither a specific instrumentation nor a measurement method can be the reason for the deviation. From *Figure 6* it is apparent that the deviations from the reference value cannot be attributed to the low radioactivity on the filters alone. It is important to note, that overestimation is more typical in the low spiked radioactivity level range (<0.2 Bq).

The presence of statistical outliers among the reported results was investigated using the Grubbs' test at a level of significance α=1% and α=5%, as suggested in ISO/IEC 5725-2 (ISO, 1994). Statistical analysis of the results was carried out for the different working dilutions (D1-D4) and for the ratio of the measured-to-reference ^{137}Cs activity separately. The reference value of the working dilutions is based on the standardised reference value of the mother solution and the dilution factors. The laboratory values used for the statistical analysis were calculated for each laboratory according to the following formula and grouped on the basis of the working dilution used (D1-D4):

$$C = \frac{A}{m} \qquad (2)$$

where

A is the activity value reported by the participating laboratory (Bq)

m is the mass of the working dilution spiked by JRC-IRMM on the surface of the air filter (g)

In the case of the ratio values according to the Grubb's test at 1% three results were indicated as outliers: Laboratory 11, 34 and 73; at 5% additionally, Laboratory 27 was indicated as outlier.

Moreover, the distribution of the data was tested using the normal probability plot and the frequency histogram. According to both of these graphs presented in *Figure 7*, the ^{137}Cs data are distributed normally and unimodally.

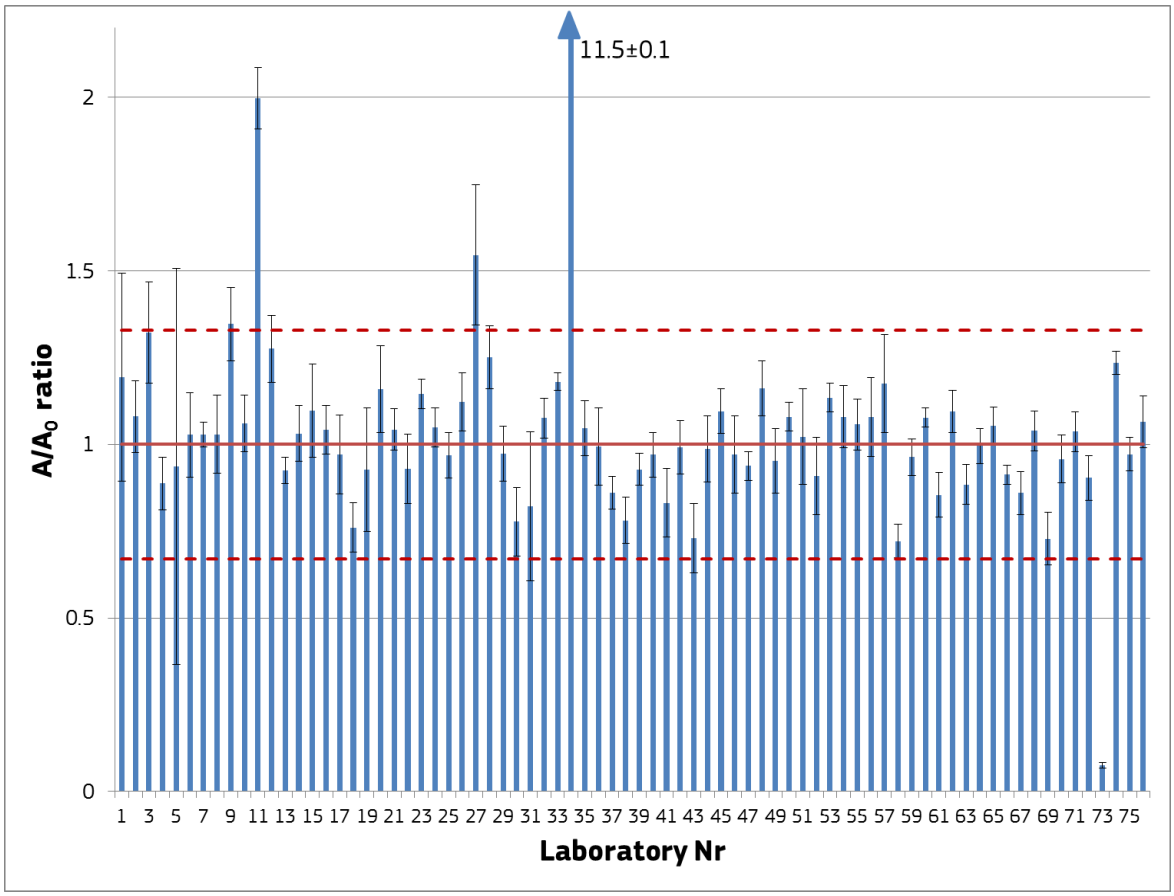

Fig. 6. Ratio of ^{137}Cs activity per filter as measured by the participating laboratory over the individual spiked activity on the filter (JRC-IRMM reference value) sorted in ascending order of the amount of spiked activity. Dashed lines indicate the 33% limit from the JRC-IRMM reference value.

The *z* values presented in *Figure 7* are calculated according to the ISO 13528:2015(E), (ISO, 2015) in the following way:

$$z = \frac{(\frac{A}{A_0} - 1)}{\sigma_{pt}} \qquad (3)$$

where

A	is the activity value reported by the participating laboratory
A_0	is the assigned activity reference value
σ_{pt}	is the standard deviation for proficiency assessment

Table 5. Statistical analysis of the laboratory reported results.

		D1	D2	D3	D4	Ratio
All reported results	Number of laboratories	7	19	28	22	76
	Min (Bq g^{-1})	9.99	0.10	0.59	0.19	0.07
	Max (Bq g^{-1})	13.66	2.55	1.08	3.01	11.47
	Median (Bq g^{-1})	10.75	1.33	0.85	0.26	1.03
	Mean (Bq g^{-1})	**11.27**	**1.35**	**0.82**	**0.38**	**1.15**
	Standard deviation (Bq g^{-1})	1.21	0.47	0.11	0.59	1.22
Omitting outliers (5%)	Number of outliers	0	3	0	1	4
	Mean (Bq g^{-1})	**11.27**	**1.35**	**0.82**	**0.26**	**1.01**
	Standard deviation (Bq g^{-1})	1.21	0.25	0.11	0.04	0.14
Omitting outliers (1%)	Number of outliers	0	2	0	1	3
	Mean (Bq g^{-1})	**11.27**	**1.35**	**0.82**	**0.26**	**1.02**
	Standard deviation (Bq g^{-1})	1.21	0.25	0.11	0.04	0.15
	Ref value (Bq g^{-1})	**11.06**	**1.28**	**0.82**	**0.26**	**1.00**
	Expanded unc. (Bq g^{-1})	0.19	0.02	0.01	0.004	0.01
	Rel. exp. unc. (%)	1.70	1.70	1.70	1.71	0.01

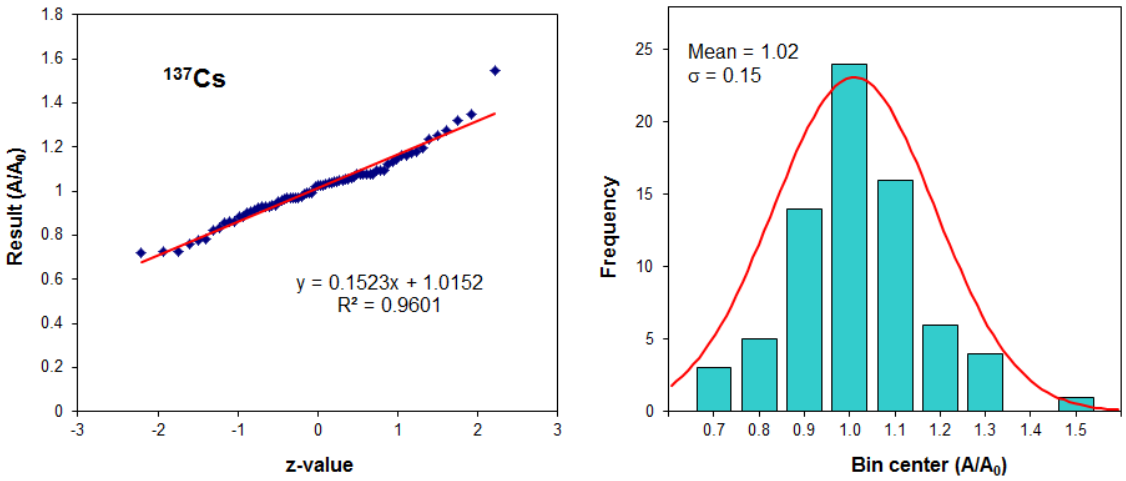

Fig. 7. Normal probability plot and frequency histogram of the ^{137}Cs results after exclusion of the three outliers (α=1%) (see text). The red curve in the frequency histogram is the normal probability distribution.

7.2 Scores and evaluation criteria

7.2.1 Percentage difference

An alternative way of presentation albeit yielding no new information versus ratio, is the percentage difference (ISO, 2005a). *Table 6* contains the percentage difference from the reference activity value calculated using the formula:

$$D_{\%} = \frac{A - A_0}{A_0} \cdot 100\% \qquad (4)$$

where

A is the reported result
A_0 is the assigned reference value

These values are plotted in ascending order in deviation chart and the laboratories reporting too low or too high values become more visible. For the environmental radioactivity measurements the criterion of ±20% difference from the reference value is usually used (*Figure 8*).

The majority of the laboratories obtained satisfactory results, nevertheless 20% of the results (15 laboratories) deviated more than 20% from the reference values. The three outlying laboratories are again clearly visible. It is also visible that the submitted results are slightly higher than the reference value. Similar trend was observed for ^{137}Cs in the case of an interlaboratory comparison on the determination of gamma emitting radionuclides in simulated air filters organised by IAEA (IAEA, 2010).

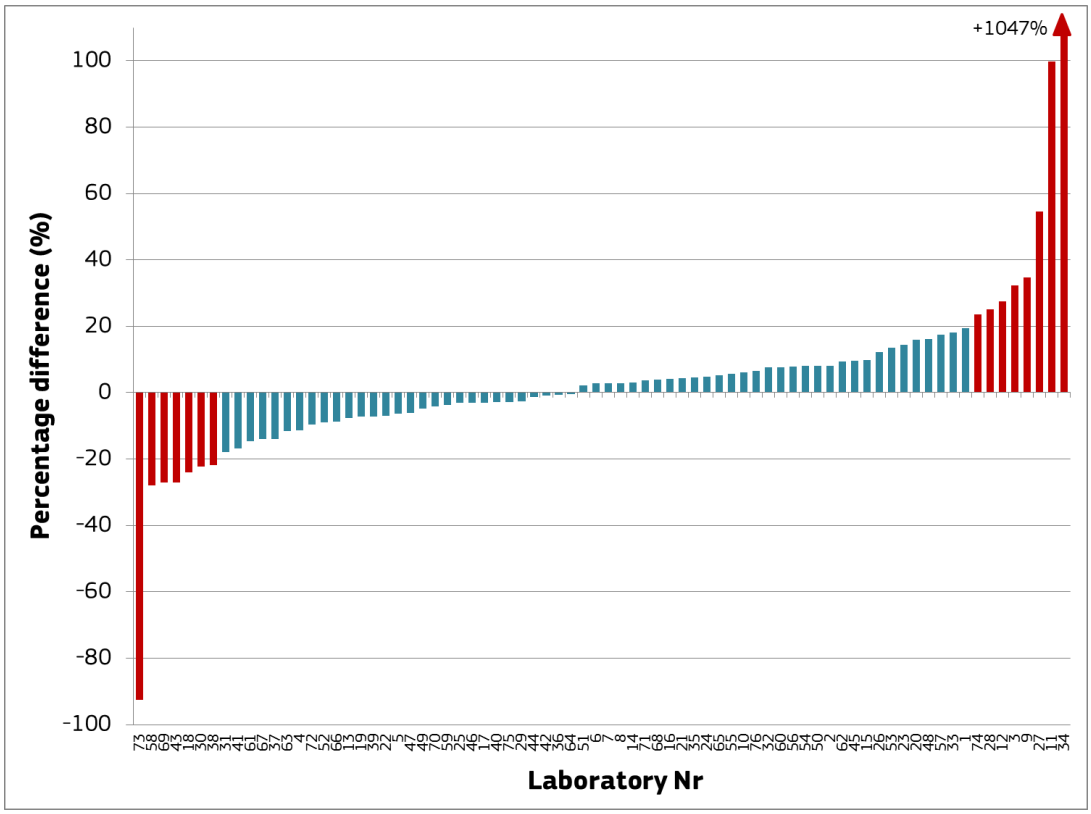

Fig. 8. Chart of the percentage difference of the results reported by the participating laboratories from the reference activity values, plotted in ascending order. Blue colour indicates the results within the range ±20% from the reference value and red indicates results outside this range.

7.2.2 E_n number

To take the expanded uncertainty of the reported results and that of the reference values into account in the analysis, a performance test using E_n numbers was applied (ISO, 2005a). The calculation of the E_n numbers was carried out according to the following formula:

$$E_n = \frac{A - A_0}{\sqrt{U(A)^2 + U(A_0)^2}} \qquad (5)$$

where

A	is the participant's results
A_0	is the assigned reference value
$U(A)$	is the expanded uncertainty of a participant's result (k=2)
$U(A_0)$	is the expanded uncertainty of the assigned reference value (k=2)

When uncertainties are estimated according to the Guide to the Expression of Uncertainty Measurement (GUM) (ISO, 2008), a measurement result with its uncertainty interval giving a level of confidence of 95% should overlap with the reference value and its expanded uncertainty. Therefore, E_n numbers are interpreted as following:

If $|E_n| \leq 1$, the laboratory values are compatible with the reference value;

If $|E_n| > 1$, the laboratory values differ significantly from the reference values, the sources of deviation should be investigated and corrected, "warning signal";

If $|E_n| > 1.5$, there is an urgent need to investigate and find the sources of the large deviation, "action signal".

The E_n numbers sorted in ascending order are graphically presented in *Figure 9*. Under the conditions of this test, 53 results out of 76 are compatible with the reference value while 23 are not. Among those 23, 12 laboratories reported incompatible results with $|E_n| > 1.5$. Comparing *Figure 8* and *Figure 9*, it is obvious that results with significant deviations from the reference value are scoring badly with E_n numbers as well. However, that comparison also shows that some laboratories with an acceptably small deviation from the reference value are assigned with an incompatible E_n number, as they probably underestimated the uncertainty on their reported value.

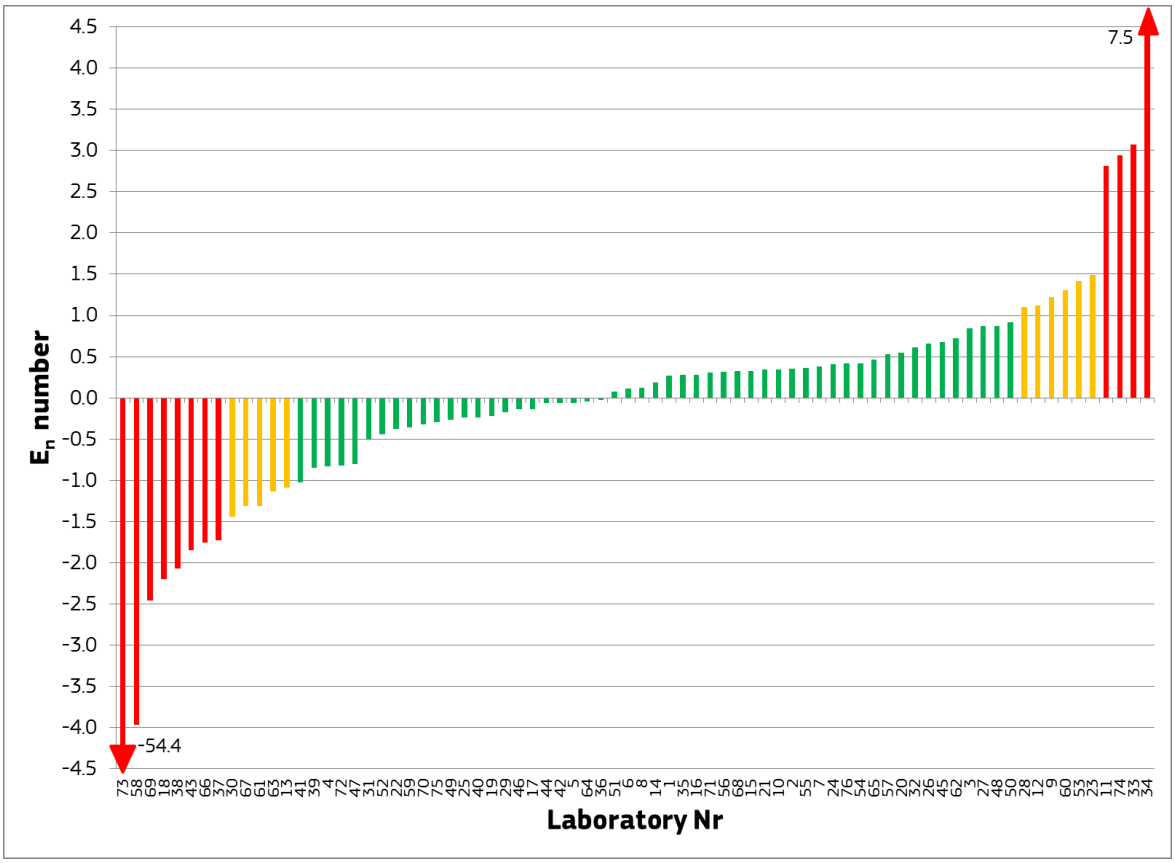

Fig. 9. E_n numbers plotted in ascending order. Green colour indicates compatible results, yellow indicates warning signal and red indicates action signal.

7.2.3 Compatibility test

The reference values, the reported values, the ratios, the percentage differences and the E_n numbers are given for each laboratory in the *Table 6*. In the case of the percentage difference the values deviating more than 20% from the reference value are indicated with yellow, the values deviating more than 33% from the reference value are indicated with bold letters. For the E_n number, the values with deviation larger than 1 are coloured orange, while the values larger than 1.5 are coloured red. The last column of *Table 6* contains the compatibility test. Results which pass both the percentage difference ($D_{\%}$) and E_n tests are considered as compatible. On the basis of the compatibility test it can be concluded that 51 laboratories (67%) provided compatible results, which is very similar to the results of the previous air filter exercise organised by JRC-IRMM in 2003.

Table 6. Comparison and compatibility test of the results reported by the 76 laboratories.

Lab Nr	A_0 (Bq)	$u(A_0)$ (Bq) k=1	A (Bq)	$u(A)$ (Bq) k=1	A/A_0	$u_c(A/A_0)$ k=1	$D_{\%}$	E_n	Compati-bility
1	0.069	0.001	0.0827	0.02475	1.19	0.30	19	0.3	yes
2	0.073	0.001	0.078812	0.0081675	1.08	0.10	8	0.4	yes
3	0.074	0.001	0.0978	0.0142	1.32	0.15	32	0.8	no

Lab Nr	A_0 (Bq)	$u(A_0)$ (Bq) $k=1$	A (Bq)	$u(A)$ (Bq) $k=1$	A/A_0	$u_c(A/A_0)$ $k=1$	$D_{\%}$	E_n	Compati-bility
4	0.074	0.001	0.066	0.005	0.89	0.08	-11	-0.8	yes
5	0.075	0.001	0.07	0.04	0.94	0.57	-6	-0.1	yes
6	0.075	0.001	0.077577	0.009435	1.03	0.12	3	0.1	yes
7	0.083	0.001	0.085	0.003	1.03	0.04	3	0.4	yes
8	0.084	0.001	0.0868	0.0098	1.03	0.11	3	0.1	yes
9	0.085	0.001	0.114	0.012	1.35	0.11	**35**	1.2	**no**
10	0.098	0.001	0.104	0.0085	1.06	0.08	6	0.3	yes
11	0.107	0.001	0.214	0.019	2.00	0.09	**100**	2.8	**no**
12	0.118	0.001	0.151	0.0145	1.28	0.10	28	1.1	**no**
13	0.119	0.001	0.11	0.004	0.92	0.04	-8	-1.1	no
14	0.121	0.001	0.125	0.01	1.03	0.08	3	0.2	yes
15	0.122	0.001	0.134	0.018	1.10	0.13	10	0.3	yes
16	0.123	0.001	0.128	0.009	1.04	0.07	4	0.3	yes
17	0.128	0.001	0.124	0.014	0.97	0.11	-3	-0.1	yes
18	0.130	0.001	0.099	0.007	0.76	0.07	-24	-2.2	**no**
19	0.132	0.001	0.12247	0.02175	0.93	0.18	-7	-0.2	yes
20	0.138	0.001	0.16	0.02	1.16	0.13	16	0.6	yes
21	0.138	0.001	0.144	0.0085	1.04	0.06	4	0.3	yes
22	0.140	0.001	0.13	0.013	0.93	0.10	-7	-0.4	yes
23	0.147	0.001	0.168	0.007	1.14	0.04	14	1.5	no
24	0.154	0.001	0.161	0.009	1.05	0.06	5	0.4	yes
25	0.158	0.001	0.153	0.01	0.97	0.07	-3	-0.2	yes
26	0.160	0.001	0.18	0.015	1.12	0.08	12	0.7	yes
27	0.163	0.001	0.252	0.051	1.55	0.20	**55**	0.9	**no**
28	0.165	0.001	0.206	0.0187	1.25	0.09	25	1.1	**no**
29	0.169	0.001	0.164	0.013	0.97	0.08	-3	-0.2	yes
30	0.170	0.001	0.132	0.013	0.78	0.10	-22	-1.4	**no**
31	0.171	0.001	0.14	0.03	0.82	0.21	-18	-0.5	yes
32	0.171	0.001	0.184	0.0105	1.08	0.06	8	0.6	yes

Lab Nr	A_0 (Bq)	$u(A_0)$ (Bq) $k=1$	A (Bq)	$u(A)$ (Bq) $k=1$	A/A_0	$u_c(A/A_0)$ $k=1$	$D_{\%}$	E_n	Compati-bility
33	0.178	0.002	0.21	0.005	1.18	0.03	18	3.1	**no**
34	0.179	0.002	2.05	0.125	11.47	0.06	**1047**	7.5	**no**
35	0.182	0.002	0.19	0.015	1.05	0.08	5	0.3	yes
36	0.185	0.002	0.184	0.0205	0.99	0.11	-1	0.0	yes
37	0.190	0.002	0.163	0.0075	0.86	0.05	-14	-1.7	**no**
38	0.192	0.002	0.15	0.01	0.78	0.07	-22	-2.1	**no**
39	0.193	0.002	0.179	0.008	0.93	0.05	-7	-0.8	yes
40	0.194	0.002	0.188	0.012	0.97	0.06	-3	-0.2	yes
41	0.196	0.002	0.163	0.016	0.83	0.10	-17	-1.0	yes
42	0.198	0.002	0.196	0.015	0.99	0.08	-1	-0.1	yes
43	0.206	0.002	0.15	0.015	0.73	0.10	-27	-1.8	**no**
44	0.215	0.002	0.212	0.02	0.99	0.09	-1	-0.1	yes
45	0.224	0.002	0.245	0.016	1.10	0.06	10	0.7	yes
46	0.242	0.002	0.235	0.026	0.97	0.11	-3	-0.1	yes
47	0.267	0.002	0.25	0.01	0.94	0.04	-6	-0.8	yes
48	0.271	0.002	0.315	0.025	1.16	0.08	16	0.9	yes
49	0.279	0.002	0.266	0.0245	0.95	0.09	-5	-0.3	yes
50	0.280	0.002	0.302	0.012	1.08	0.04	8	0.9	yes
51	0.284	0.002	0.29	0.04	1.02	0.14	2	0.1	yes
52	0.297	0.003	0.27	0.03	0.91	0.11	-9	-0.4	yes
53	0.298	0.003	0.338	0.014	1.13	0.04	13	1.4	no
54	0.315	0.003	0.34	0.03	1.08	0.09	8	0.4	yes
55	0.322	0.003	0.34	0.025	1.06	0.07	6	0.4	yes
56	0.417	0.004	0.45	0.1	1.08	0.11	8	0.3	yes
57	0.426	0.004	0.5	0.07	1.18	0.14	18	0.5	yes
58	0.441	0.004	0.318	0.015	0.72	0.05	-28	-4.0	**no**
59	0.488	0.004	0.47	0.025	0.96	0.05	-4	-0.4	yes
60	0.529	0.005	0.57	0.015	1.08	0.03	8	1.3	no
61	0.553	0.005	0.4723	0.0303	0.85	0.06	-15	-1.3	no

Lab Nr	A_0 (Bq)	$u(A_0)$ (Bq) $k=1$	A (Bq)	$u(A)$ (Bq) $k=1$	A/A_0	$u_c(A/A_0)$ $k=1$	$D_{\%}$	E_n	Compati-bility
62	0.597	0.005	0.654	0.039	1.09	0.06	9	0.7	yes
63	0.599	0.005	0.53	0.03	0.88	0.06	-12	-1.1	no
64	0.603	0.005	0.6	0.03	1.00	0.05	0	0.0	yes
65	0.617	0.005	0.65	0.035	1.05	0.05	5	0.5	yes
66	0.619	0.005	0.565	0.0145	0.91	0.03	-9	-1.8	**no**
67	0.664	0.006	0.571	0.035	0.86	0.06	-14	-1.3	no
68	0.674	0.006	0.7	0.04	1.04	0.06	4	0.3	yes
69	0.677	0.006	0.493	0.037	0.73	0.08	-27	-2.5	**no**
70	0.839	0.007	0.803	0.0549	0.96	0.07	-4	-0.3	yes
71	1.119	0.010	1.16	0.065	1.04	0.06	4	0.3	yes
72	1.206	0.010	1.09	0.07	0.90	0.06	-10	-0.8	yes
73	1.323	0.011	0.099	0	0.07	0.01	**-93**	-54.4	**no**
74	1.411	0.012	1.7424	0.055	1.23	0.03	23	2.9	**no**
75	1.502	0.013	1.46	0.07	0.97	0.05	-3	-0.3	yes
76	2.310	0.020	2.46	0.18	1.06	0.07	6	0.4	yes

7.2.4 Possible influencing parameters

Based on the results reported by the participants and answers provided to the questionnaire *Table 7* contains the detailed evaluation of possible influencing parameters to the measurement performance and therefore $D_{\%}$ and E_n number. The correlation between laboratories with eventually less experience (mostly only authorised without accreditation, certification, <25 samples annually and first time participating in EC ILCs) and higher ratio of incompatible results is evident. No strong correlation can be found between the air filter size, spiked activity level and sample preparation and the deviation of the submitted result from the reference value. Regarding the E_n number, the highest number of incompatible results with E_n>1.5 is observed in the case of the large-size air filters. In the case of laboratories which did not follow their routine conditions or could not follow their routine conditions the correct estimation of uncertainty was more problematic. Regarding the counting efficiency calibration the results show the importance of using suitable high quality reference sources.

Table 7. Statistics and categorisation of the reported results by the participating laboratories. Possible influencing parameters, where the number of results per category is high enough for statistical evaluation, are indicated in red.

		Total Nr of labs	$D_{\%}$>20 (Nr of labs)	Ratio (%)	E_n>1 (Nr of labs)	Ratio (%)
All participants		76	15	**20**	23	**30**
Country	EU	68	12	18	20	29
	Non-EU	8	3	38	3	38
Accreditation	Accredited	49	7	14	14	29
	Authorised	28	10	36	10	36
	Certified (ISO 9000)	12	0	0	2	17
Participation in EC ILCs	First EC ILC	15	5	33	6	40
	2003 - ^{137}Cs in air filters	29	4	14	8	28
	All EC ILCs on ^{137}Cs measurements	16	3	19	4	25
^{137}Cs measurement per year	<25	17	5	29	7	41
	25-100	31	5	16	8	30
	>100	27	6	22	8	26
Air filter size	Small	32	8	25	8	25
	Medium	14	2	14	3	21
	Large	30	5	17	12	40
Spiked activity level (Bq)	0.07-0.1	10	2	20	1	10
	0.1-0.2	32	8	25	11	34
	0.2-0.3	11	1	9	2	18
	0.3-1	17	2	12	7	41
	>1	6	2	33	2	33
Routine conditions	Yes	60	12	20	17	28
	No	16	3	19	6	38
Measurement of plastic foil	Together with air filter	29	8	28	9	31

		Total Nr of labs	$D_{\%}$>20 (Nr of labs)	Ratio (%)	E_n>1 (Nr of labs)	Ratio (%)
	Air filter alone	47	7	15	14	30
Sample preparation	No sample preparation	26	7	27	7	27
	Pressing/ compressing	21	3	14	7	33
	Folding	20	2	10	5	25
	No information provided	11	0	0	1	9
	Packing with blank filters	4	1	25	2	50
	Cutting	2	2	100	2	100
	Milling	1	0	0	1	100
	Burning	1	1	100	1	100
Efficiency calibration	Calibration source	30	3	10	7	23
	Dedicated software	21	3	14	7	33
	Reference air filter	20	6	30	9	45
	No information provided	8	2	25	1	13
	No calibration	1	1	100	1	100
Reported uncertainty budget	Consistent	41	7	17	12	29
	Inconsistent	35	8	23	11	31
Notes/ problems	Inhomogeneity	6	1	17	2	33
	Routinely more air filter, for EC ILC the spiked sample alone	6	1	17	1	17
	Geometry	7	1	14	4	57

7.2.5 PomPlot

In order to compare the results, a modern type of graph, the "PomPlot", which underlines the importance of the assigned uncertainties is applied. The theoretical description of the PomPlot can be found in *Annex 13*.

The PomPlot created on the basis of the results omitting the laboratories which reported erroneous results is presented in *Figure 10*. The many points outside the |ζ|=1 and 2, and the few outside |ζ|=3, indicate that laboratories underestimated the uncertainties.

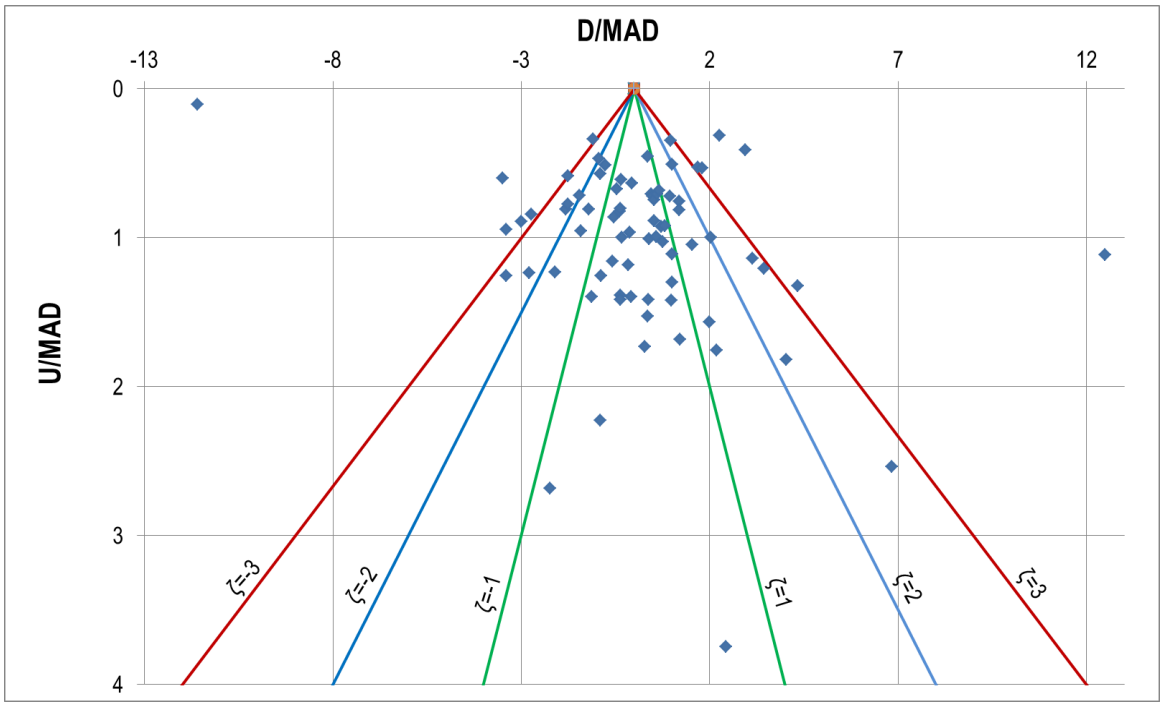

Fig. 10. PomPlot of the ^{137}Cs data. Green, blue and red solid lines indicate ζ-scores=1, 2 and 3, respectively.

8 Comparison of the 2003 and the 2014 EC ILC

To compare the submitted results of the current EC ILC with the EC ILC organised in 2003 it can be generally concluded that the measurement performance of the laboratories was slightly improved, since 87.5% of the laboratories reported within ±33% interval of the reference value in 2003, while 93% of the laboratories reported within ±33% interval of the reference value in the current EC ILC. However the estimation of the uncertainty is a critical point in the case of the reporting of results. More information on the EC ILCs organised in 2003 and 2014 can be found in the *Table 8*.

Thirty laboratories participated in both air filter EC ILCs and, since one laboratory measured 2 air filters in 2003, reported 31 results in 2003 and 30 results in 2014. Nine laboratories improved their performance since they were outliers and/or in the incompatible E_n number groups in the 2003 exercise and reported compatible values in 2014. Two laboratories which were in action level regarding E_n number provided values in warning level, show a slightly improvement. However 7 laboratories which reported compatible values in 2003 are outliers and/or reported results giving incompatible E_n numbers in 2014; this could be an indication that the quality of the results provided by these laboratories is not stable over time (*Figure 11*), or alternatively, are overconfident on the accuracy of their results.

Table 8. Comparison of the spiked activity levels and the performance of the laboratories in the 2003 and 2014 EC ILC.

	2003	**2014**	**Laboratories Participated in both ILC**	
			2003	**2014**
Number of air filters	48	76	30	
Spiked ^{137}Cs activity levels in Bq				
Minimum	0.015	0.070	0.029	0.083
Maximum	0.564	2.340	0.401	2.310
Average	0.160	0.359	0.162	0.324
Median	0.123	0.195	0.126	0.194
Outliers in number of air filters				
Ratio > ±33%	6 (13%)	5 (7%)	2 (7%)	2 (7%)
$D_{\%}$ > ± 20%	18 (38%)	15 (20%)	6 (20%)	4 (13%)
E_n > 1	3 (6%)	11 (14%)	3 (10%)	6 (20%)
E_n > 1.5	9 (19%)	12 (16%)	6 (20%)	2 (7%)

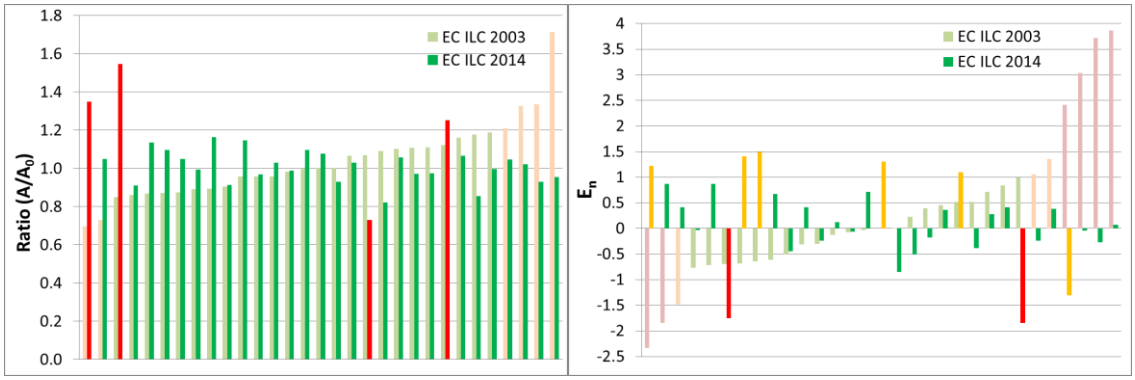

Fig. 11. Comparison of the performance of laboratories in 2003 and 2014.

9 Conclusion

In the 2014 EC ILC on the determination of ^{137}Cs in air filters organised by JRC-IRMM 76 laboratories from EU and non-EU countries participated. The answers given to the questionnaires revealed a diversity of sampling devices and sampling procedures, leaving much room for variation in the measurement geometry and sample preparation of air filters. If just one type of air filter with the same activity would have been sent to the participating laboratories, a comparison of results would not have reflected the routine measurement conditions.

On the basis of the submitted results it can be stated that almost all of the laboratories could report reliable measurement results. Only 7% (5 laboratories) of the laboratories reported activity values outside of ±33% range of the reference value. In the case of 3 laboratories the source of the deviation is known, and one laboratory is just over the limit with 35% deviation from the reference value. However, the largest deviations from the reference value could not be attributed to the low level of ^{137}Cs activity or to a specific sample preparation for measurement scheme alone. On the basis of E_n number, which takes into account the reported result and the reference value uncertainty as well, 30% of the participants reported incompatible results, of which 11 laboratories are in the warning range and 12 laboratories are in the action zone. A possible reason could be that the participants did not follow the "GUM approach", with the consequence of an incomplete evaluation of uncertainty sources. A workshop was organised by JRC-IRMM on April 7, 2016, to bring together the participants of this EC ILC to discuss all aspects of the ILC and provide ideas and suggestions to improve the performance of the laboratories.

The fact that some problems are always encountered which can be detected and corrected confirms that there is a permanent need for such comparisons to reaffirm the performance of the laboratories. The Metrology for radiological early warning networks in Europe (ENV57-MetroERM) project, coordinated in the frame of European Metrology Research Programme and funded by the European Association of National Metrology Institutes (EURAMET) and the EU in 2014, aims as well to the harmonisation of the data provided by the radiological early warning networks in Europe – the largest and most comprehensive environmental radiation monitoring system worldwide (MetroERM, 2015). This means the harmonisation of procedures for dose rate and activity concentration measurements with the development and validation of novel and improved instrumentation, traceable calibration procedures, new dose rate and contamination level calculation procedures with background level calculations and appropriate correction methods, and novel traceable reference materials and standard sources will be promoted. In the frame of the EMRP ENV57/MetroERM a new interlaboratory comparison exercise was organised by the JRC-IRMM in February 2016.

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10 ANNEXES

Annex 1: List of participating laboratories (in alphabetical order)

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Mr Johannes Klimstein
AGES Linz
Department of Radon and Radioecology
Wieningerstraße 8
4020 Linz

Mr Florian Smecka
AGES Vienna
Radiation Protection and Radiochemistry
Spargelfeldstraße 191
1220 Vienna

AZERBAIJAN

Mr Faig Mutallimov
The Ministry of Ecology and Natural Resources
National Environmental Monitoring Department
Haydar Aliyev avenue 50
AZ1033 Baku

BELGIUM

Mr Tony Dieudonne
IRE ELIT
BUS
Avnue de l'Esperance 1
6220 Fleurus

Mrs Liesel Sneyers
SCK•CEN
Low Radioactive Measurements
Boeretang 200
2400 Mol

BOSNIA-HERZEGOVINA

Mrs Delveta Deljkic
Institute for Public Health of Federation of Bosnia and Herzegovina
Radiation Protection Centre
Tahtali sokak 17
71 000 Sarajevo

BULGARIA

Mrs Kitka Dimitrova
Executive Environment Agency
Regional Laboratory Varna
Jan Palah str. 4
9000 Varna

Mrs Bistra Hristova
Regional Health Inspectorate - Varna
Public Health Department
Bregalnitsa str. 3
9000 Varna

Mrs Rositza Kamenova-Totzeva
National Center of Radiobiology and Radiation Protection
Rad. Cont. Dep., Public Exp. Mon. Lab.
Georgi Sofiiski Blvd. 3
1606 Sofia

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Executive Environment Agency, Ministry of Environment and Water
Radioactivity Measurement Laboratory Department
Exarh Josif str. 81
3000 Vratza

Mr Mihail Shishenkov
Executive Environment Agency, Ministry of Environment and Water
Radioactivity Measurement Laboratory Department
Tzar Boris III Blvd. 136
1618 Sofia

CROATIA

Dr Tomislav Bituh
Institute for Medical Research and Occupational Health
Radiation Protection Unit
Ksaverska cesta 2
10000 Zagreb

CYPRUS

Mrs Anastasia Caballero
State General Laboratory of Cyprus
Radioactivity Lab of Food and Environmental samples
Kimonos Str 44
1451 Nicosia

CZECH REPUBLIC

Mr Zdenek Borecky
Statni ustav radiacni ochrany, v.v.i.
Branch Hradec Kralove
Pileticka 57
500 03 Hradec Kralove

Mr Jiri Rada
Statni ustav radiacni ochrany,v.v.i.
Syllabova 21
703 00 Ostrava

Dr Petr Rulík
National Radiation Protection Institute (SÚRO)
Monitoring Department
Bartoškova 28
140 00 Prague

Mrs Eva Šindelková
State Office for Nuclear Safety (SUJB)
Regional centre SUJB Ceske Budejovice
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Technical University of Denmark
Center for nuclear technologies, Nutech
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Mr Asser Poulsen
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Miljoelab
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2730 Herlev

ESTONIA

Mrs Eia Jakobson
Environmental Board Republic of Estonia
Radiation Safety Department
Kopli 76
10416 Tallinn

FINLAND

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STUK - Radiation and Nuclear Safety Authority
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Laippaite 4
00880 Helsinki

FRANCE

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Bat 501, Bois des Rames
91400 Orsay

GERMANY

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SW 2.5 / Atmosphärische Radioaktivität und Süurenanalyse
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79098 Freiburg

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TI24
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63067 Offenbach

GREECE

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HUNGARY

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OSSKI
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Radioanalytical Reference Laboratory
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Environmental Protection Agency
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ITALY

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Mr Michael Froggatt
Cavendish Nuclear Limited
Analytical Services
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CA24 3HZ Whitehaven

Annex 2: Traceability to BIPM SIR

BIPM, F-92310 SEVRES

International Reference System for the Activity Measurement of Gamma-Ray Emitting Nuclides (SIR)

Radionuclide: ^{137}Cs Half life adopted: $T_{1/2}$ = (11 020.8 ± 1.2) d

				Data reported by laboratory				Ionization-chamber measurements carried out at BIPM		
								Activity A_e which would produce the same ion current as the Ra source		
Labora-tory	Ampoule number	Method of standardisation	Reference date	Activity at ref. date (kBq)	Rel. uncert. Category A (r_1,%)	Rel. uncert. Category B (r_2,%)	Date	Relative uncertainty (r_3, %)	A_e (kBq)	Combined uncert. of A_e $A_e(r_1^2 + r_2^2 + r_3^2)^{1/2}$ (kBq)
IRMM	137Cs0310	4πβ-NaI(Tl)γ and efficiency tracing with ^{134}Cs and LS counting (CIEMAT/NIST method)	2003-07-01	**980.8**	**0.59**	0.0	2004-01-20	**0.093**	**27 337**	**163**

The key comparison reference value (KCRV) for ^{137}Cs has been identified as: **27 549 ± 44 kBq**

(BIPM comparison BIPM.RI (II)-K1.Cs-137 of activity measurements of the radionuclide ^{137}Cs and links for the 1982 international comparison CCRI(II)-K2Cs-137,G. Ratel, C. Michotte, BIPM 2003/07/17)

Annex 3: Communication to the participants

3A. Invitation letter for nomination

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Nuclear Safety, Security and Safeguards

Ref. Ares(2014)2977196 - 11/09/2014

Geel, 11 September 2014
JRC.D.4/TA/mvdl/

Mesdames	**M. J. Bacao Madruga (PT)**	**Messrs.**	**A. Abramenkovs (LV)**
	H. Halachliyska (BG)		**K. G. Andersson (DK)**
	R. Kamenova-Totzeva (BG)		**P. Andersson (SE)**
	S. Krca (HR)		**E. Bedi (SK)**
	M. Lecomte (LU)		**P. Brajda (MT)**
	M. Lepasson (EE)		**M. Cindro (SI)**
	C. McGuire (UK)		**J. Claes (BE)**
	L. Peake (UK)		**J. J. Diana (FR)**
	S. Quell (LU)		**C. Katzlberger (AT)**
	M. R. Salas (ES)		**K. Kelleher (IE)**
	E. Simion (RO)		**P. Kwakman (NL)**
	V. Starostova (CZ)		**F. Lepriéur (FR)**
	B. Vilimaite-Silobritiene (LT)		**P. Lipinski (PL)**

A. Malitezos (EL)
G. Manificat (FR)
G. Menna (IT)
J. Peter (DE)
A. Polt (EE)
S. Runaeres (UK)
L. Sombre (BE)
R. Stapel (DE)
G. Torri (IT)
M. Tzortzis (CY)
K. Vesterbacka (FI)
A. Vincze (HU)
M. Yeomans (MT)

Subject: Articles 35-36 of the Euratom Treaty
Nomination of laboratories for the EC interlaboratory comparison on Cs-137 measurement in air filters

Dear colleague,

As you know, EU Member States are obliged under Art. 35 and 36 of the EURATOM Treaty (and as further specified in the Commission Recommendation 2000/473/EURATOM) to inform the European Commission (EC) on a regular basis of the radioactivity levels in their environment. In order to obtain more information on the measurement methods and on the quality of the values reported by the Member States, the EC (DG JRC on request of DG ENER) organises regularly a European interlaboratory comparison exercise.

After discussions with DG ENER.D.4 and during the Euratom Treaty Art. 35-36 meetings, the JRC IRMM is preparing to organise an interlaboratory comparison exercise on **Cs-137 measurement in air filters**. A similar exercise was organised in 2003 and the aim of the present interlaboratory comparison is to obtain an overview of the measurement methods applied by the participating laboratories and of any improvements, which occurred in the meantime.

The schedule for the interlaboratory comparison is as follows: A first questionnaire to request blank filters and information for spiking them individually for each laboratory will be sent in October 2014. The spiked filters are expected to be dispatched to the participating laboratories in early December 2014. The participating laboratories will be requested to send their results to JRC IRMM by the end January 2015. The preliminary report is foreseen to be available in May 2015.

It would be appreciated if you could investigate which laboratories in your country would be interested in participating in this exercise or which laboratories you would like to see participating. To proceed according to the plans, we require your (nationally coordinated) response by **3 October 2014**.

Due to the limited availability of human resources, the exercise must be curtailed to two laboratories per country. If you should wish to nominate more than two laboratories, exceptions might be possible depending on the total number of participants.

We request you to provide us with your nationally coordinated answer, containing the contact data (responsible person, complete mail address, telephone, telefax and e-mail) for the nominated laboratories with an indication of priority for your country, should you nominate more than 2 laboratories.

Please, send your replies to the functional e-mail box
mailto:JRC-IRMM-REM-COMPARISONS@ec.europa.eu

Looking forward to hearing from you with the laboratory nominations, I remain,

Yours sincerely,

Timotheos Altzitzoglou
ILC-REM Project Leader

cc Messrs. Ivo Alehno, Vesa Tanner, Alan Ryan (DG ENER.D.3, Luxembourg)
Mr. Marc De Cort (JRC ITU-Ispra)
Messrs. Willy Mondelaers, M. Hult, Ms. Borbála Máté (JRC IRMM)

2

Retiseweg, B-2440 Geel - Belgium, Telephone: +32 14 571 211 Fax: +32 14 584 273
Telephone: direct line +32 14 571 251, +32 14 571 266
E-mail: Timotheos.ALTZITZOGLOU@ec.europa.eu, Borbala.MATE@ec.europa.eu
Internet: https://ec.europa.eu/jrc/en/institutes/irmm/

Ref. Ares(2014)3397696 - 14/10/2014

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Nuclear Safety, Security and Safeguards

Geel, 14 October 2014
JRC.D.4/TA/BM/ARES

Subject: **Invitation to the EC interlaboratory comparison on Cs-137 measurement in air filters**

Dear colleague,

EU Member States are obliged under Art. 35 and 36 of the Euratom Treaty (and as further specified in Commission Recommendation 2000/473/Euratom) to inform the European Commission (EC) on a regular basis on the radioactivity levels in their environment, in some food products and in drinking water. In order to obtain more information on the quality of the values reported by the Member States, the EC (through its Directorate General JRC) organises regularly a European interlaboratory comparison exercise.

Your laboratory has been nominated by your national representative(s) or authority to participate in the above mentioned interlaboratory comparison organised by JRC-IRMM.

In order to provide you with air filter spiked with the appropriate activity level, similar to what you measure routinely, and allow you to measure following your usual procedure and measurement geometry, we kindly ask you to reply to a questionnaire on sampling method, air filter used and measurement conditions. The questionnaire you can find at:

http://ec.europa.eu/eusurvey/runner/ICS-REM2014

The password is: ICSREM2014

You are also requested to send us two (2) new (unused, blank) air filters to the attention of:

Mrs. Borbála MÁTÉ
European Commission
Directorate-General Joint Research Centre
Institute for Reference Materials and Measurements
Retieseweg 111
B-2440 Geel, Belgium

Please, reply to the questionnaire and send the blank air filters as soon as possible, but no later than **5 November 2014**.

Once we receive your answers to the questionnaire and the blank filters, we will give you instructions on how to register online for the Interlaboratory Comparison. The spiked samples with further instructions will be sent to the address specified in the registration

Retieseweg, B-2440 Geel - Belgium. Telephone: +32 14 571 211 Fax: +32 14 584 273
Telephone: direct line +32 14 571 251, +32 14 571 266

E-mail: JRC-IRMM-REM-COMPARISONS@ec.europa.eu
Internet: https://ec.europa.eu/jrc/institutes/irmm/

form. The spiked filters are expected to be dispatched to the participating laboratories in early December 2014. The participating laboratories will be requested to send their results to JRC IRMM by the end of January 2015. The reporting of the results will be done via a web-based questionnaire. The preliminary report of the exercise is foreseen to be available in May 2015.

Should you have any question, please feel free to contact us at:

Email: JRC-IRMM-REM-COMPARISONS@ec.europa.eu

Looking forward to hearing from you, we remain,

Yours sincerely,

Timotheos Altzitzoglou
ICS-REM Project Leader

cc Messrs. Ivo Alehno, Vesa Tanner, Alan Ryan (DG ENER.D.3, Luxembourg)
Mr. Marc de Cort (JRC ITU-Ispra)
Mesdames Beatriz de la Calle, Borbala Mate (JRC IRMM)
Messrs. Willy Mondelaers, Pieter Dehouck, Mikael Hult (JRC IRMM)

3C. Registration (email)

Subject: EC interlaboratory comparison on Cs-137 measurement in air filters – registration

Dear colleague,

We received your blank air filters and submitted questionnaire. We are now in the phase of spiking the filters with ^{137}Cs.

The reporting of laboratory results will be done via Internet. Therefore, we kindly ask you to register your laboratory via the following WEB link:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1322

Please be aware that deadline for the registration is 05 January 2015!

Should you have any question, please feel free to contact us at:

Email: JRC-IRMM-REM-COMPARISONS@ec.europa.eu

Looking forward to hearing from you, we remain,

Yours sincerely,

Timotheos ALTZITZOGLOU — ICS-REM Project Leader

Borbala MATE — ICS-REM Project Coordinator

3D. Spiked air filter dispatch (email) and accompanying letter

Subject: EC interlaboratory comparison on Cs-137 measurement in air filters – spiked air filter dispatch

Dear colleague,

The parcel containing the spiked air filter was dispatched to your laboratory by DHL courier from our site (IRMM).

Please confirm the receipt of the sample by e-mail to JRC-IRMM-REM-COMPARISONS@ec.europa.eu. Please check the spiked air filter and in case of any damage report to the above e-mail address.

If you have already confirmed the receipt of the sample you don't have to confirm it again.

The parcel contains:

a) Test item: spiked air filter

b) Accompanying letter

The reporting of the results is done via the login page using the following URL: https://web.jrc.ec.europa.eu/ilcReportingWeb

To report your results you need a password key which is unique to this interlaboratory comparison and your laboratory. You will find your password key in the accompanying letter. Keep the accompanying letter for further reference!

The deadline for reporting results and completing the questionnaire is Friday, 27 February 2015.

Should you have any question, please do not hesitate to contact us.

We wish you success with your measurements.

Kind regards,

Timotheos ALTZITZOGLOU — ICS-REM Project Leader

Borbala MATE — ICS-REM Project Coordinator

Ref. Ares(2015)214709 - 20/01/2015

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate D - Institute for Reference Materials and Measurements
Standards for Nuclear Safety, Security and Safeguards

Geel, 20 January 2015
JRC.D.4/TA/bm/ARES(2015)

«Title» «First_Name» «Surname»
«Company»
«Department»
«Street_Name»
«Zip_Code» «City»
«Country»

Participation in the EC interlaboratory comparison on ^{137}Cs measurement in air filters

Dear «Title» «Surname»,

Thank you for participating in the EC interlaboratory comparison on ^{137}Cs measurement in air filters.

Enclosed, you will find one of your blank filters spiked with ^{137}Cs activity either at a level you normally measure or higher than the threshold you indicated in the questionnaire.

Please confirm the receipt of the sample by e-mail to JRC-IRMM-REM-COMPARISONS@ec.europa.eu. Please check the spiked air filter and in case of any damage report to the above e-mail address.

This parcel contains:
a) Test item: spiked air filter
b) This accompanying letter.

Please keep this letter; you will need it to report your results.

On the filter you can easily distinguish the active spots (light blue colour). This should make further preparation of the filter for measuring easier. Large filters had to be folded; the activity is on the inner side.

For the measurement and analysis, you should follow the procedure you routinely use. In case you do not measure the filter in the inner plastic bag, it is advisable to measure also the bag to make sure that no activity from the filter is left in the plastic bag.

Reporting of the results:
The reporting of the results is done via the login page using the following URL: https://web.jrc.ec.europa.eu/ilcReportingWeb

Retieseweg, B-2440 Geel - Belgium. Telephone: (32-14)571-211.
Office: 010 00/005B. Telephone: direct line (32-14)571-251.

E-mail: JRC-IRMM-REM-COMPARISONS@ec.europa.eu

To report your results you need a password key which is unique to this interlaboratory comparison and your laboratory.

Your password key is: «Part_key»

Please note that only **submitted** results will be taken into account, therefore, do not only *Save* your results but also click on the ***Submit*** button. Once you have submitted your results and questionnaire, please remember to send us a signed copy by e-mail (JRC-IRMM-REM-COMPARISONS@ec.europa.eu) or to fax us (Fax no. +32 14 584 273).

As you have been informed, the description of your analytical and measurement procedures will be collected via questionnaire using the same URL link as for reporting the results. We kindly ask you to answer all relevant questions regarding the procedures you employed for the measurement of the filter sample. Disregard questions which are not related to the methods used in your laboratory. The uncertainty of the result must be reported in the same units as the activity concentration (**Bq**).

Please, notice that during the reporting of your results the *Cancel* button serves as an exit or return button.

The deadline for reporting results and completing the questionnaire is **Friday, 27 February 2015.**

Should you have any question, please do not hesitate to contact us.

We wish you success with your measurements.

Kind regards,

Timotheos ALTZITZOGLOU
ICS-REM Project Leader

3E. Information for participants during the measurement exercise (emails)

Subject: EC interlaboratory comparison on Cs-137 measurement in air filters – reference date

Dear colleague,

As several of you asked for the reference date for this exercise, this is set for 01/01/2015 0:00 UTC.

This information is also available in the online reporting system.

For your calculation we recommend to use the half-life of Cs-137 proposed by the Decay Data Evaluation Program (DDEP):

30.05 ± 0.08 years* or 10975 ± 29 days*

* http://www.nucleide.org/DDEP_WG/DDEPdata.htm

Should you have any question, please do not hesitate to contact us.

Kind regards,

Timotheos ALTZITZOGLOU — ICS-REM Project Leader

Borbala MATE — ICS-REM Project Coordinator

Subject: EC interlaboratory comparison on Cs-137 measurement in air filters – reporting of results

Dear colleague,

We would like to make you aware that, as mentioned in the sample accompanying letter, both the activity result and its uncertainty should be reported in the unit bequerel (Bq), (not mBq or %).

The reporting of the results is done via the login page using the following URL: https://web.jrc.ec.europa.eu/ilcReportingWeb

To report your results you need a password key, which is unique to this interlaboratory comparison and your laboratory, delivered to you with the sample.

We kindly remind you that the deadline for reporting results and completing the questionnaire is Friday, 27 February 2015.

Should you have any question, please do not hesitate to contact us.

Kind regards,

Timotheos ALTZITZOGLOU — ICS-REM Project Leader

Borbala MATE — ICS-REM Project Coordinator

3F. Communication of preliminary results

Subject: EC interlaboratory comparison on Cs-137 measurement in air filters – preliminary results

Dear «Title» «Family_name»,

First of all, thank you for your participation in the 2014 EC interlaboratory comparison on ^{137}Cs measurement in air filters. Currently, we are working on the evaluation of the results for the preparation of the final report. However, for your information we are sending you a preliminary evaluation of the results of this comparison in the form of a graph. Anonymity being a requirement, each laboratory was assigned a new code number (different than the original one you received with the spiked filter).

The new code number for your laboratory is «Lab_code».

You can identify your laboratory in the graph and find out how your reported result compares to the reference value.

In Figure 1, the ratio of the ^{137}Cs activity per filter as reported by the participating laboratories over the individual spiked activity on the filter (JRC-IRMM reference value) for each filter sorted in ascending order of the laboratory code number is shown. The uncertainties of the ratio values are the combined uncertainties of the measured and the reference values (k=1). Dashed lines indicate the ±33% deviation from the JRC-IRMM reference value. Reference date is 2015-01-01 0:00 UTC.

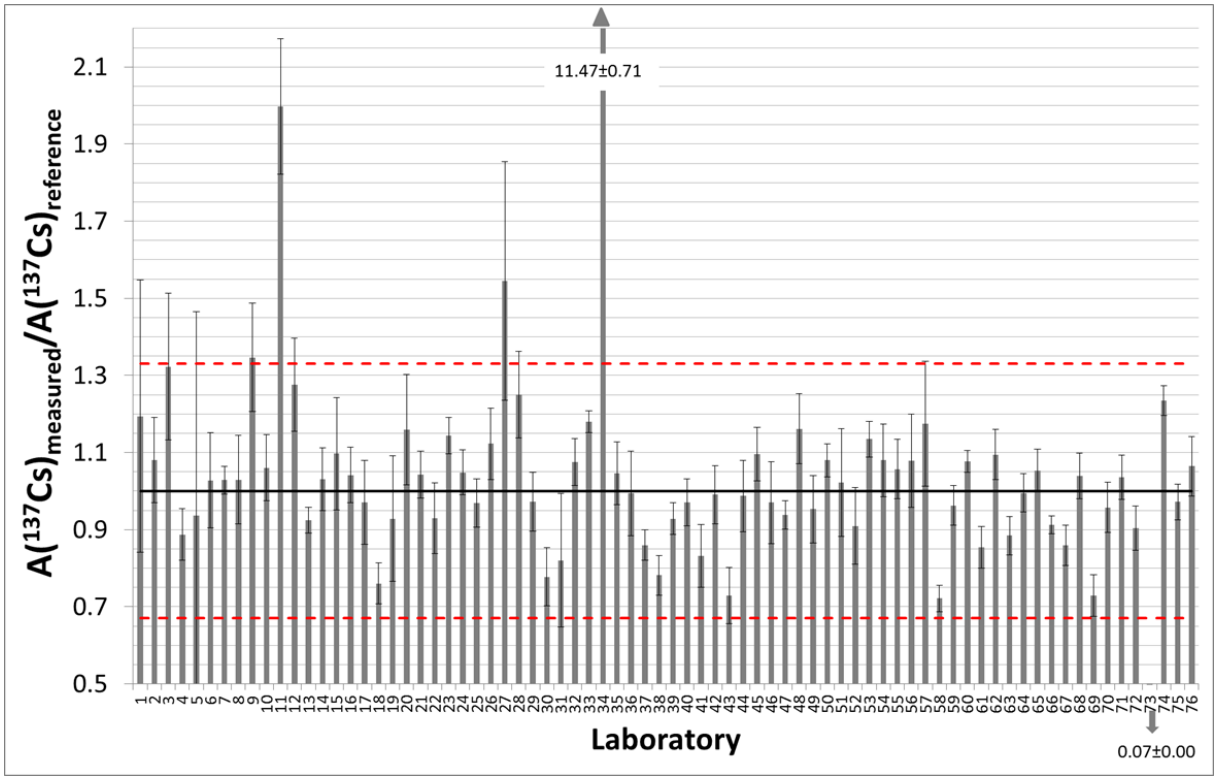


Figure 1. Ratio of the ^{137}Cs activity per filter as reported by the participating laboratory over the individual spiked activity on the filter

The final report of this comparison exercise is foreseen to be available later in 2015.

If you have any further questions with respect to this comparison, please feel free to contact us at JRC-IRMM-REM-COMPARISONS@ec.europa.eu .

Sincerely yours,

Timotheos ALTZITZOGLOU — ICS-REM Project Leader

Borbala MATE — ICS-REM Project Coordinator

Annex 4: Questionnaire sent to participants to collect information on air filters and sampling

Questionnaire for participants ICS-REM 2014

Fields marked with * are mandatory.

1 Organisation and contact details

1.1 Person details

1.1.1 Title
- Mr
- Mrs
- Dr

1.1.2 First Name*

1.1.3 Family Name*

1.1.4 Email*

1.1.5 Gender*
- Male
- Female

1.1.6 Telephone

1.1.7 Extension

1.1.8 Fax

1.2 Organisation details

1.2.1 Organisation*

1.2.2 Organisation Department

1.2.3 Organisation Address*

1.2.4 Zip code*

1.2.5 City*

1.2.6 Country*

1.2.7 Mailing address (to which the filter will be sent back):*
- Same as organisation address
- Different than organisation address

1.2.8 If it different than the organisation address, please provide the complete mailing address here:*

1.2.9 Telephone

1.2.10 Extension

1.2.11 Fax

3.1 Sampling: provide the following parameters for the air sampling method

3.1.1 Supplier and model of air sampler:*

3.1.2 Volume flow rate (m3/h):

3.1.3 Type of flow measurement and control, if any:

3.1.4 Calibration of flow measurement

3.1.5 Total air volume sampled (m3/filter):*

3.1.6 Sampling period (hours) and frequency (eg. once a week):*

3.2 Air filters: define the following information on the material used for the air filters

3.2.1 Supplier and type of filter material:*

3.2.2 Shape and size of filter:*

3.2.3 How many measurements (Cs-137 in air filters) do your laboratory perform per year?

- < 25
- 25-100
- > 100

3.2.4 Blank weight (areal density - mg/cm2):

1.2.12 E-mail

2 Laboratory

2.1 What is the type of your laboratory (more than one choice is possible)?*

- Research and development
- Radioactivity in the environment
- Monitoring of nuclear facilities
- Fissile material control or safeguards
- Governmental laboratory
- University laboratory
- Other

2.2 If other, please specify here:

2.3 Is your laboratory certified, accredited or authorised (more than one choice is possible)?*

- Certified (ISO 9000)
- Accredited
- Authorised

2.4 If accredited, please provide the details for which radionuclides/methods are you accredited:

2.5 In which ICS-REM (EC ILCs) did you participate previously (more than one choice is possible)?*

- This ILC is the first one in which we participate
- 2003 - Cs-137 in air filters
- 2005 - Cs-137, K-40 and Sr-90 in milk powder
- 2008 - Ra-226, Ra-228, U-234 and U-238 in mineral waters
- 2010 - Radionuclides in soil
- 2011 - K-40, Sr-90 and Cs-137 in bilberry powder
- 2012 - Gross alpha and beta activity in mineral waters

2.6 If the name of the organisation/laboratory is changed since the previous ILC(s), please indicate the previous name(s) here:

3 Measurement

3.2.5 Typical aerosol deposit collected (mg/cm2):

3.2.6 Specify the transport of filters between sampling station and measurement laboratory:

3.2.7 Typical activity level measured for Cs-137 (indicate unit as well):*

3.2.8 Would you like to get the filters spiked with coloured solution in order to optically identify the active spots?*

- ○ Yes
- ○ No

3.2.9 Specific the requirements for the spiked filters:*

- ☐ Cs-137 activity level
- ☐ Pattern
- ☐ Packaging
- ☐ Other
- ☐ No special requirement

3.2.10 Specify the required Cs-137 activity level in Bq:*

3.2.11 Specify the required pattern:*

3.2.12 Specify the required packaging:*

3.2.13 Specify here the other requirements:*

3.3 Sample preparation and measurements

3.3.1 What type of detector is used for the determination of Cs-137 in air filters?*

- ☐ Ge(Li) detector
- ☐ HPGe detector
- ☐ BEGe detector
- ☐ Well type detector
- ☐ NaI(Tl) detector
- ☐ Other

3.3.2 If other, please specify here:

3.3.3 Describe briefly the measurement methods, with sample preparation if any:*

3.3.4 How often are the calibration and background/blank measurements carried out?

3.3.5 Provide the Cs-137 specific, typical background level of your instrument and the detection limit of your routine method for the Cs-137 isotope:

4 Additional information

4.1 Do you determine any additional radionuclide(s) besides Cs-137 in the air filters?

- ○ Yes
- ○ No

4.2 If yes, please specify the radionuclide(s) here:

4.3 Describe the difficulties encountered:

4.4 Further comments on sampler, filter, sampling, measurements etc.:

Annex 5: Responses of the participants to the questionnaire for collecting information on air filters and sampling

To determine the % ratios in *Table A-1* and the subsequent tables, the 76 participants were taken as 100%.

Table A-1. General information on the 2014 EC ILC participating laboratories.

Question	Number of answers	Ratio (%)
"What is the type of your laboratory (more than one choice is possible)?"		
Research and development	23	30
Radioactivity in the environment	71	93
Monitoring of nuclear facilities	20	26
Fissile material control or safeguards	2	3
Governmental laboratory	38	50
University laboratory	9	12
Other	2	3
"Is your laboratory certified, accredited or authorised (more than one choice is possible)?"		
Certified (ISO 9000)	12	16
Accredited	49	65
Authorised	28	37
"In which ICS-REM (EC ILCs) did you participate previously (more than one choice is possible)?"		
This ILC is the first one in which we participate	15	20
2003 - ^{137}Cs in air filters	30	40
2005 - ^{137}Cs, ^{40}K and ^{90}Sr in milk powder	29	38
2008 - ^{226}Ra, ^{228}Ra, ^{234}U and ^{238}U in mineral waters	23	30
2010 - Radionuclides in soil	34	45
2011 - ^{40}K, ^{90}Sr and ^{137}Cs in bilberry powder	44	58
2012 - Gross alpha and beta activity in mineral waters	36	47
"At how many ICS-REM (EC ILCs) did you participate previously?"		
This EC ILC is the first one in which we participate	15	20
All 6 EC ILCs	9	12
In 5 EC ILCs	10	13
In 4 EC ILCs	7	9
In 3 EC ILCs	7	9
In 2 EC ILCs	14	18
In 1 EC ILC	14	18

Table A-2. Air filter size categories.

Size group	Size of air filter	Number of air filters	Ratio (%)
small	Ø 4.7-13 cm	32	42
intermediate	18x20-23.5x28 cm	14	18
large	24.6x41-60x70 cm	30	40

Table A-3. Information on the air sampling methods.

Question	Answer		
	Range	**Average**	**Median**
Small size group			
Total volume of air sampled/filter (m^3)	1.2-28800	1705	100
Sampling period (h)	1-400	58	24
Sampling frequency	Mostly on daily basis		
Intermediate size group			
Total volume of air sampled/filter (m^3)	1200-60000	18186	8500
Sampling period (h)	24-720	168	144
Sampling frequency	Mostly on a weekly basis		
Large size group			
Total volume of air sampled/filter (m^3)	112.5-300000	97652	100000
Sampling period (h)	24-240	153	168
Sampling frequency	Mostly on a weekly basis		

Table A-4. Information on the technical part of the measurements.

Question	Number of answers	Ratio (%)
"How many measurements (^{137}Cs in air filters) does your laboratory perform per year?"		
< 25	17	22
25-100	31	41
> 100	27	36
"Would you like to get the filters spiked with coloured solution in order to optically identify the active spots?"		
Yes	62	82
No	14	18
"Specify the requirements for the spiked filters:"		
^{137}Cs activity level - requested level varied between 0.02-1500 Bq, median: 5 Bq	38	50
Pattern - requests for homogeneous distribution, active area	19	25
Packaging - special folding, sealed bag/holder requirements	11	15
Other - air filter's side to spike, active area, folding	4	5
No special requirement	34	45
"What type of detector is used routinely for the determination of ^{137}Cs in air filters?"		
Ge(Li) detector	2	3
HPGe detector	66	87
BEGe detector	14	18
Well type detector	4	5
NaI(Tl) detector	1	1
Other	1	1
"Do you determine any additional radionuclide(s) besides ^{137}Cs in the air filters?"		
Yes	70	92
No	2	3

Annex 6: Type, material, size and shape of air filters used by the participants

Table A-5. Type, material size and shape of air filters used by the participants.

Supplier and type of filter material	Filter size, shape (cm)
Glass fibre	ø 4.7
Nitrocellulose	ø 4.7
Bernard Dumas cellulose	ø 4.7
F&J FP-47M	ø 4.7
Gelman Sciences glass fibre	ø 4.7
Hi-Q Environmental Products borosilicate glass fibre	ø 4.7
Millipore glass fibre	ø 4.7
Schleicher & Schuell glass fibre	ø 4.7
Whatman glass fibre	ø 4.7
Glass fibre	ø 4.8
F&J, FP-50M	ø 5
Whatman glass fibre	ø 5
RADECO glass fibre	ø 5.5
Whatman glass fibre	ø 5.5
HB5773 från Hollingsworth & Vose	ø 6
Whatman	ø 7
Whatman	ø 10
F&J, FP-102M2	ø 10.2
FILTER-LAB quartz microfibre	ø 10.2
3M polypropylene	ø 13
Estonian FPP-15	18 x 20
Glass fibre	20 x 25
Hi-Q Environmental Products glass fibre	20 x 25
Munktell glass fibre	20 x 25
Whatman glass fibre	20.3 x 25.2
NUPORE glass fibre	20.3 x 25.4
SKC glass fibre	20.3 x 25.4

Supplier and type of filter material	Filter size, shape (cm)
STAPLEX glass fibre	20.3 x 25.4
Whatman glass fibre	20.3 x 25.4
IREMA micro 2000plus EU5 - EU9	21 x 27
AS ESFIL TEHNO FPM 1530	23 x 28
Whatman glass fibre	23 x 28.5
AS ESFIL TEHNO FPM 1530	23.5 x 28
Camfil glass fibre	24.6 x 41
TECNASA Petrianov G3	40 x 40
HEPA-5300	40 x 60
Russian Petrianov FPP-15-1.5	41 x 41
PTI Petrianov FPP-15-1.5	42 x 42
Whatman GF/A	42 x 53
PTI Petrianov FPP-15-1.5	44 x 44
Russian Petrianov FPP-15-1.5	44 x 44
Petrianov G3	44 x 46
Glass fibre	45 x 45
PTI Petrianov G3	45 x 56
TECNASA Petrianov G3	45.5 x 45.5
Whatman glass fibre	46 × 57
3M	46 x 57
F853 BMF20	46 x 57
AS ESFIL TEHNO, IFMP 1545	46 x 57
Petrianov PTI	50 x 60
Hollingsworth & Vose HB5773	56 x 56
Petrianov	60 x 60
PTI Petrianov PPP-15-1.5	60 x 60
TECNASA Petrianov G3	60 x 60
Russian Petrianov FPP-15-1.5	60 x 70
Sterile Gauze	65 x 65

Annex 7: Sequence of spiked filters and quality control sources prepared for the EC ILC 2014 exercise.

Table A-6. Sequence of spiked filters and quality control sources prepared for the EC ILC 2014 exercise.

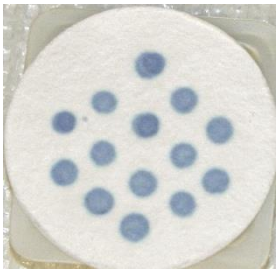
Sample ID	Source Type	Solution
B1	Dilution	A1 (mother solution)
PS_01	Point Source	A1
PS_02	Point Source	A1
LS_01	LSC source	A1
LS_02	LSC source	A1
LS_03	LSC source	A1
LS_04	LSC source	B1
LS_05	LSC source	B1
LS_06	LSC source	B1
LS_07	LSC source	B1
LS_08	LSC source	B1
C1	Dilution	B1
LS_09	LSC source	B1
LS_10	LSC source	B1
PS_03	Point Source	B1
LS_11	LSC source	B1
LS_12	LSC source	B1
PS_04	Point Source	B1
LS_13	LSC source	B1
PS_05	LSC source	B1
LS_14	LSC source	B1
LS_15	LSC source	C1
LS_16	LSC source	C1
LS_17	LSC source	C1
D1	Dilution	C1
LS_18	LSC source	C1
PS_06	Point Source	C1
D2	Dilution	C1
LS_19	LSC source	C1
D3	Dilution	C1
LS_20	LSC source	C1
PS_07	Point Source	C1
LS_21	LSC source	C1
D4	Dilution	C1
PS_08	Point Source	C1
LS_22	LSC source	C1
D4b	Dilution	C1
Filter_008	Filter	D1
Filter_047	Filter	D1
Filter_060	Filter	D1
Filter_062	Filter	D1
Filter_073	Filter	D1

Sample ID	Source Type	Solution
Filter_025	Filter	D1
Filter_029	Filter	D1
LSD_01	LSC source	D1
IRMM_01	IRMM Filter	D1
PS_09	Point Source	D1
LSD_02	LSC source	D1
IRMM_02	IRMM Filter	D1
PS_10	Point Source	D1
LSD_03	LSC source	D1
Filter_002	Filter	D2
Filter_017	Filter	D2
Filter_028	Filter	D2
Filter_030	Filter	D2
Filter_031	Filter	D2
Filter_041	Filter	D2
Filter_066	Filter	D2
IRMM_03	IRMM Filter	D2
LSD_04	LSC source	D2
LSD_05	LSC source	D2
Filter_049	Filter	D2
Filter_051a	Filter	D2
Filter_052	Filter	D2
PS_11	Point Source	D2
Filter_053	Filter	D2
Filter_056	Filter	D2
Filter_065	Filter	D2
LSD_06	LSC source	D2
Filter_069	Filter	D2
Filter_072	Filter	D2
Filter_013	Filter	D2
LSD_07	LSC source	D2
Filter_018	Filter	D2
PS_12	Point Source	D2
Filter_039	Filter	D2
Filter_064	Filter	D2
IRMM_04	IRMM Filter	D2
PS_13	Point Source	D2
PS_18	Point Source	D2
Filter_051b	Filter	D2
Filter_045	Filter	D3
Filter_058	Filter	D3
Filter_057	Filter	D3
Filter_076	Filter	D3

Sample ID	Source Type	Solution
Filter_043	Filter	D3
IRMM_05	IRMM Filter	D3
Filter_042	Filter	D3
Filter_033	Filter	D3
Filter_009	Filter	D3
Filter_015	Filter	D3
Filter_019	Filter	D3
Filter_034	Filter	D3
LSD_08	LSC source	D3
PS_14	Point Source	D3
Filter_014	Filter	D3
Filter_040	Filter	D3
Filter_071	Filter	D3
LSD_09	LSC source	D3
Filter_026	Filter	D3
Filter_048	Filter	D3
IRMM_06	IRMM Filter	D3
Filter_005	Filter	D3
Filter_016	Filter	D3
Filter_004	Filter	D3
Filter_024	Filter	D3
Filter_012	Filter	D3
Filter_023	Filter	D3
Filter_036	Filter	D3
Filter_038	Filter	D3
Filter_032	Filter	D3
Filter_022	Filter	D3
Filter_055	Filter	D3
PS_15	Point Source	D3
Filter_054	Filter	D3
Filter_035	Filter	D4
Filter_003	Filter	D4
Filter_006	Filter	D4
PS_16	Filter	D4
Filter_007	Filter	D4
Filter_010	Filter	D4
IRMM_07	IRMM Filter	D4
Filter_021	Filter	D4
LSD_10	LSC source	D4
Filter_068	Filter	D4
Filter_011	Filter	D4
LSD_11	LSC source	D4
PS_17	Point Source	D4
Filter_020	Filter	D4
IRMM_08	IRMM Filter	D4
Filter_027	Filter	D4
LSD_12	LSC source	D4
Filter_067	Filter	D4
Filter_063	Filter	D4
Filter_061	Filter	D4

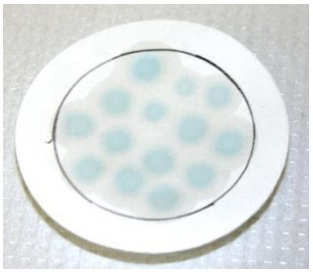
Sample ID	Source Type	Solution
Filter_059	Filter	D4
Filter_070	Filter	D4
Filter_074	Filter	D4
Filter_075	Filter	D4
Filter_046	Filter	D4
Filter_001	Filter	D4
Filter_037	Filter	D4
Filter_050	Filter	D4
Filter_044	Filter	D4

Annex 8: Examples of spiked filters.

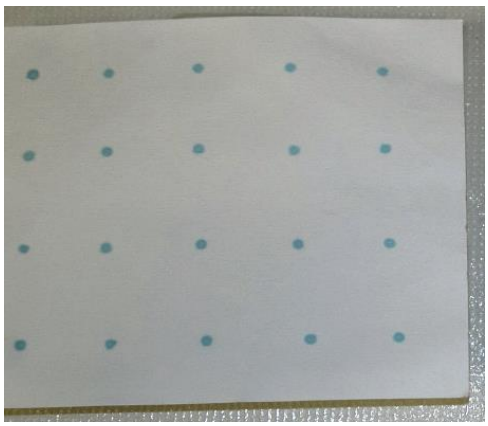


a)

b)

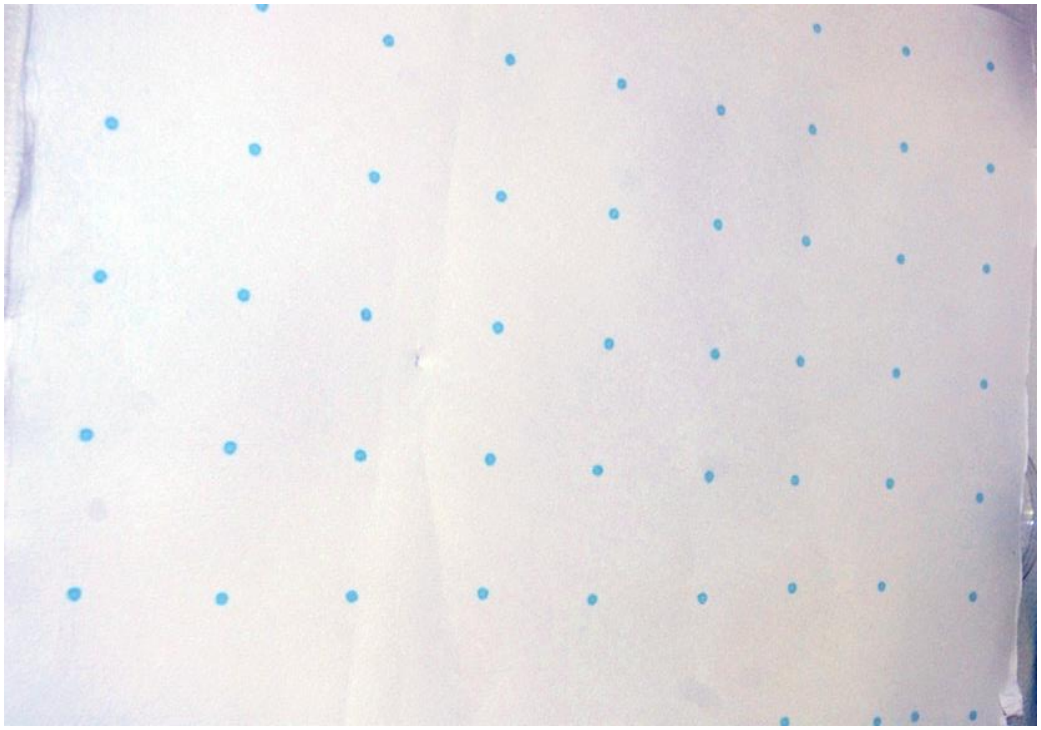


c)



d)

e)



f)

Annex 9: Quality control sources and filters; measurement results and their deviation from the reference activity values.

Table A-7. Gamma-ray spectrometric results of the quality control point sources, their relative deviation from the reference values and their ratio to the reference values.

Point Sources		Detector A					Detector B				
Sample ID	Dilution	Measured activity (Bq)	Uncer-tainty (Bq) (k=1)	Deviation from reference value (%)	Activity Ratio (Measured to Reference)	Uncer-tainty (k=2)	Measured activity (Bq)	Uncer-tainty (Bq) (k=1)	Deviation from reference value (%)	Activity Ratio (Measured to Reference)	Uncer-tainty (k=2)
PS_01	A1	23 869	207	-1.7	0.98	0.03	24 093	219	-0.8	0.99	0.03
PS_02	A1	65 085	565	-1.0	0.99	0.02	-	-	-	-	-
PS_03	B1	463	4	-1.4	0.99	0.02	468	4	-0.3	1.00	0.02
PS_04	B1	1 348	12	-0.3	1.00	0.02	1 347	12	-0.4	1.00	0.02
PS_05	B1	Not measured					Not measured				
PS_06	C1	-	-	-	-	-	5.46	0.08	0.2	1.00	0.03
PS_07	C1	6.2	0.1	-0.2	1.00	0.04	6.25	0.06	1.1	1.01	0.03
PS_08	C1	-	-	-	-	-	6.36	0.06	0.3	1.00	0.03
PS_09	D1	-	-	-	-	-	0.55	0.02	-1.9	0.98	0.08
PS_10	D1	0.443	0.017	0.2	1.00	0.04					
PS_11	D2	-	-	-	-	-	0.064	0.006	-5.1	0.95	0.18
PS_12	D2	0.042	0.009	16.0	1.16	0.53	0.036	0.001	0.9	1.01	0.08
PS_13	D3	-	-	-	-	-	0.021	0.002	-0.7	0.99	0.21
PS_14	D3	0.061	0.007	2.8	1.03	0.24	-	-	-	-	-
PS_15	D3	-	-	-	-	-	0.040	0.002	-15.0	0.85	0.10
PS_16	D4	-	-	-	-	-	0.027	0.002	-2.7	0.97	0.12
PS_17	D4	0.029	0.005	30.5	1.31	0.23					
PS_18	D2	-	-	-	-	-	0.049	0.003	0.7	1.01	0.14

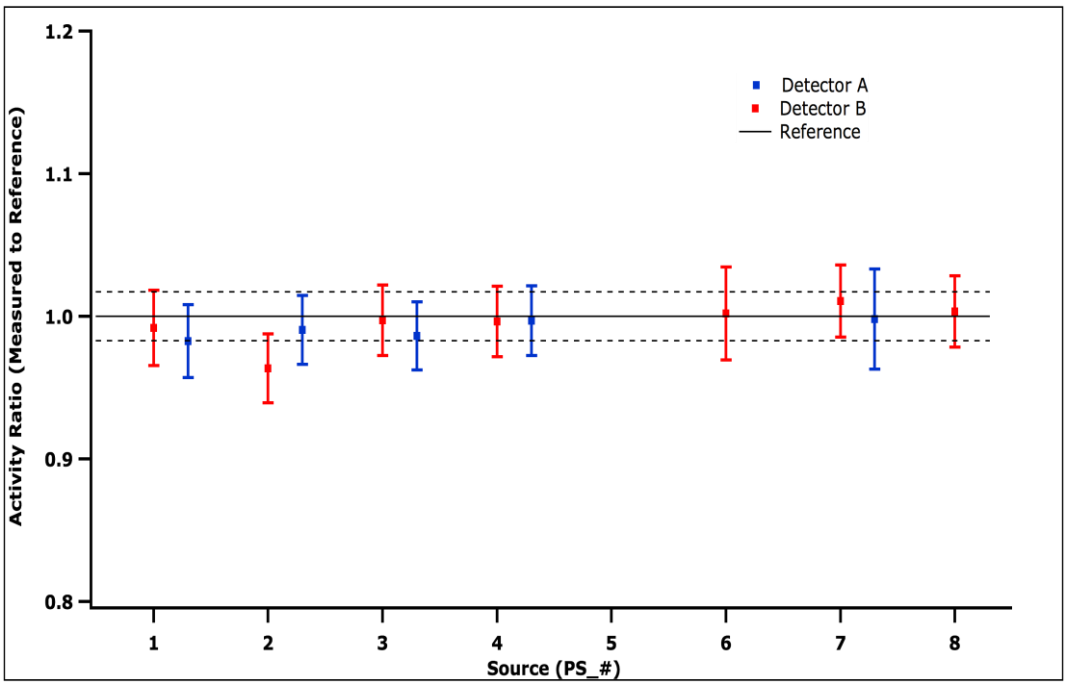


Fig. A-1. Ratio of measured-to-reference activity and expanded uncertainty (k=2) of the quality control point sources prepared from the ^{137}Cs mother solution (A1) and the dilutions B1 and C1.

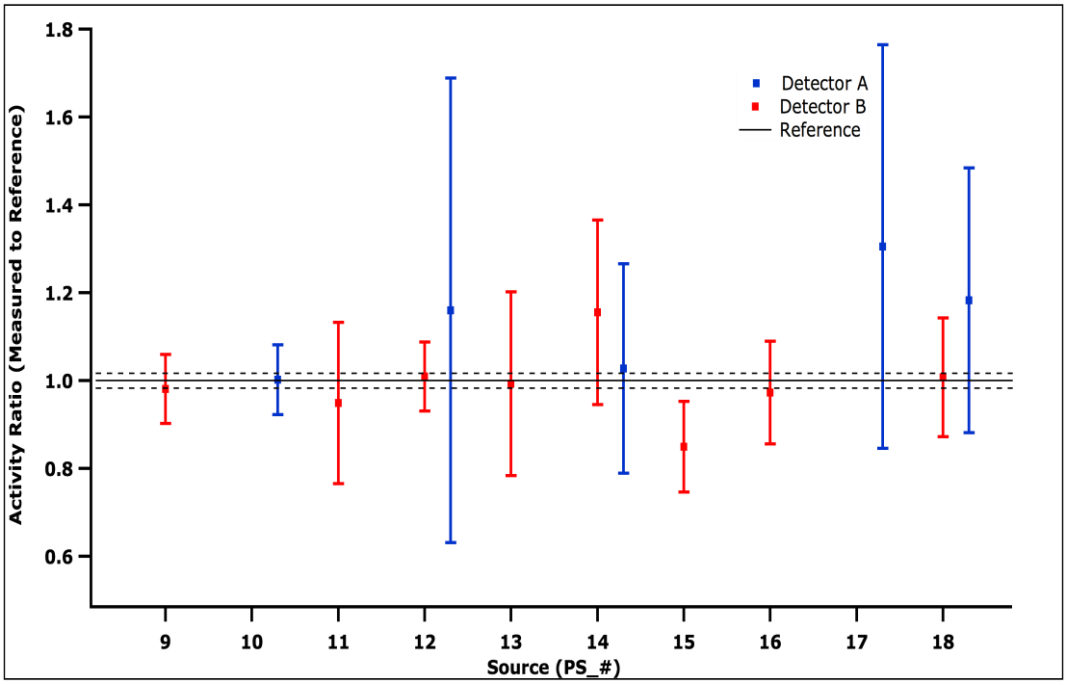


Fig. A-2. Ratio of measured-to-reference activity and expanded uncertainty (k=2) of the quality control point sources prepared from the ^{137}Cs dilutions D1 to D4.

Table A-8. Liquid Scintillation Counting (LSC) results of the quality control spiked air filters (mother solution A1 and dilutions B1 and C1), their relative deviation from the reference values and their ratio to the reference values.

Sample ID	Dilution	Measured activity (Bq)	Uncertainty (Bq) (k=1)	Deviation from expected activity (%)	Ratio	Unc (k=2)
LS_001	A1	3.59E+06	3.9E+03	-0.15	0.998	0.02
LS_002	A1	3.59E+06	3.8E+03	-0.16	0.998	0.02
LS_003	A1	3.58E+06	2.8E+02	-0.41	0.996	0.02
LS_004	B1	36 572	10	-0.14	0.999	0.02
LS_005	B1	36 627	7	0.01	1.000	0.02
LS_008	B1	36 601	5	-0.06	0.999	0.02
LS_009	B1	36 613	5	-0.03	1.000	0.02
LS_010	B1	36 614	8	-0.02	1.000	0.02
LS_011	B1	36 624	6	0.00	1.000	0.02
LS_012	B1	36 589	7	-0.09	0.999	0.02
LS_013	B1	36 624	9	0.01	1.000	0.02
LS_014	B1	36 632	10	0.03	1.000	0.02
LS_015	C1	276.5	1.1	0.10	1.001	0.02
LS_016	C1	274.7	0.7	-0.53	0.995	0.02
LS_017	C1	277.2	1.2	0.36	1.004	0.02
LS_018	C1	277.1	1.1	0.31	1.003	0.02
LS_019	C1	248.5	1.5	-10.0	0.900	0.02
LS_020	C1	263.9	1.6	-4.5	0.955	0.02
LS_021	C1	263.6	1.6	-4.6	0.954	0.02
LS_022	C1	230.7	5.1	-16.5	0.835	0.04

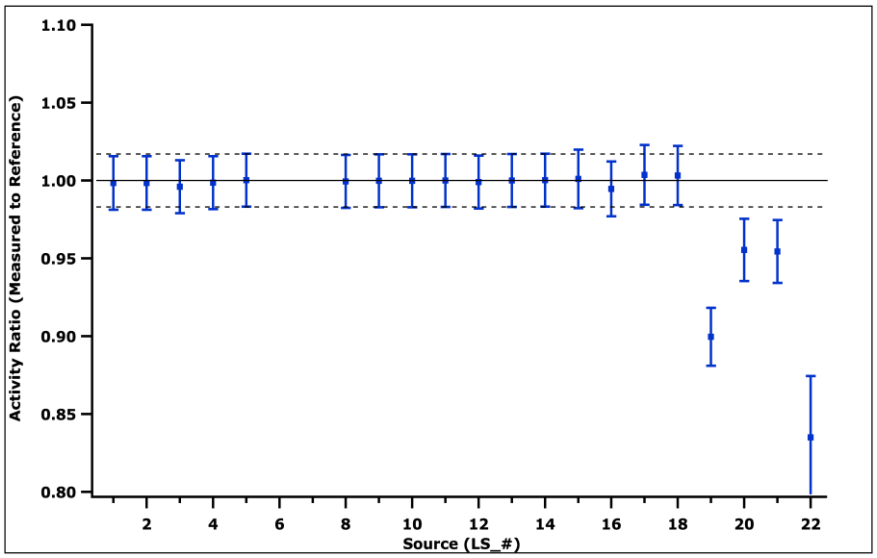


Fig. A-3. Ratio of measured-to-reference activity and expanded uncertainty (k=2) of the quality control LSC sources prepared from the ^{137}Cs mother solution A1 and the dilutions B1 and C1.

Table A-9. Liquid Scintillation Counting (LSC) results of the quality control spiked air filters (dilutions D1 to D4), their relative deviation from the reference values and their ratio to the reference values.

Sample ID	Dilution	Measured activity (Bq)	Uncertainty (Bq) (k=1)	Deviation from expected activity (%)	Ratio	Unc (k=2)
LS_D001	D1	11.15	0.06	0.80	1.008	0.02
LS_D002	D1	10.95	0.04	-1.01	0.990	0.02
LS_D003	D1	11.06	0.06	-0.05	1.000	0.02
LS_D004	D2	1.18	0.01	-7.3	0.927	0.02
LS_D005	D2	1.22	0.03	-4.7	0.953	0.05
LS_D006	D2	1.38	0.04	8.4	1.084	0.06
LS_D007	D2	1.18	0.03	-7.6	0.924	0.04
LS_D008	D3	0.75	0.03	-8.0	0.920	0.07
LS_D009	D3	0.80	0.03	-1.9	0.981	0.08
LS_D010	D4	0.20	0.05	-22	0.776	0.38
LS_D011	D4	0.27	0.04	2.0	1.020	0.31
LS_D012	D4	0.21	0.07	-19	0.811	0.53

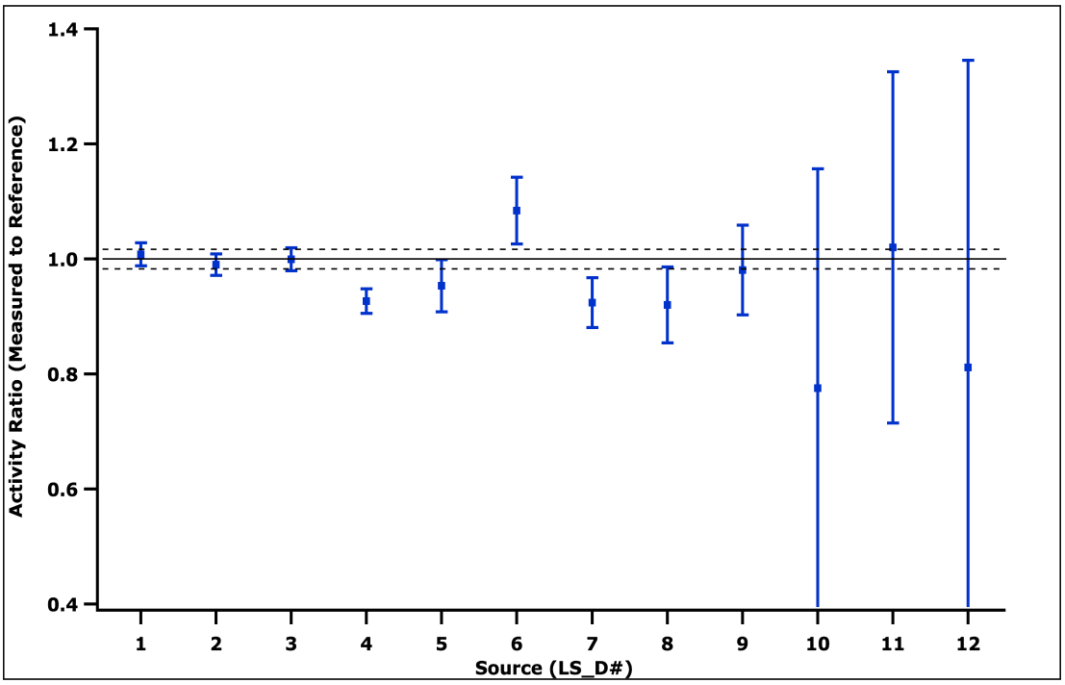


Fig. A-4. Ratio of measured-to-reference activity and expanded uncertainty (k=2) of the quality control LSC sources prepared from the ^{137}Cs dilutions D1 to D4.

Table A-10. Gamma-ray spectrometric results of the quality control spiked air filters, their relative deviation from the reference values and their ratio to the reference values.

Filters		Detector A					Detector B				
Sample ID	Dilution	Measured activity (Bq)	Uncer-tainty (Bq) (k=1)	Deviation from reference value (%)	Activity Ratio (Measured to Reference)	Uncer tainty (k=2)	Measured activity (Bq)	Uncer-tainty (Bq) (k=1)	Deviation from reference value (%)	Activity Ratio (Measured to Reference)	Uncer-tainty (k=2)
IRMM001	D1	0.47	0.02	-9.5	0.90	0.08	0.51	0.02	-0.9	0.99	0.08
IRMM002	D1	0.71	0.03	1.8	1.02	0.09	0.68	0.01	-2.5	0.97	0.04
IRMM003	D2	0.15	0.01	-9.2	0.91	0.16	0.152	0.007	-6.9	0.93	0.09
IRMM004	D2	0.122	0.006	-6.7	0.93	0.10	0.145	0.005	10.5	1.10	0.08
IRMM005	D3	0.062	0.005	-9.1	0.91	0.13	-	-	-	-	-
IRMM006	D3	0.081	0.004	2.6	1.03	0.11	-	-	-	-	-
IRMM007	D4	0.043	0.003	0.6	1.01	0.13	-	-	-	-	-
IRMM008	D4	0.033	0.003	-0.7	0.99	0.18	-	-	-	-	-

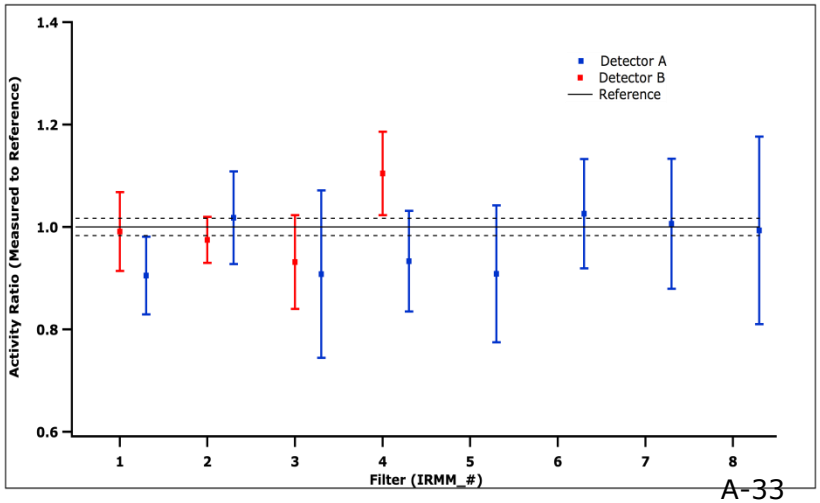


Fig. A-5. Ratio of measured-to-reference activity and expanded uncertainty (k=2) of the quality control spiked air filters, prepared from the ^{137}Cs dilutions D1 to D4.

Mlc questionnaire

Comparison for Reporting of the results for Cs-137 in air filters

As it was previously announced, the reporting of your measurement method and results will be done via an online questionnaire. We kindly ask you to answer all relevant questions regarding the procedures you employed for the measurement of the filter sample. Disregard questions which are not related to the methods used in your laboratory. Questions marked with asterisk (*) are mandatory to answer. Please, be aware that the deadline for the submission of the results and the questionnaire is Friday, 27 February 2015. Thank you for your cooperation!

Submission Form

1. **Sample treatment**

1.1. Were the comparison sample treated according to the same analytical procedure as routinely used in your laboratory for the same type of samples (Cs-137 in air filters)? *

- a) Yes
- b) No

1.1.1. If not, please specify the differences here: *

1.2. Did you measure the sample together with the plastic foil or did you measure the foil separately? *

- a) Together
- b) Separately
- c) The plastic foil was not measured

1.2.1. If separately, please provide the measured activity of the plastic foil: *

1.3. Did you apply any preconcentration or chemical treatment? *

- a) Yes
- b) No

1.3.1. If yes, please describe the method (chemicals and procedures used, chemical recovery obtained, method of chemical recovery determination, etc.): *

1.4. Describe the method used for the source preparation (geometry, type of equipment used, etc.): *

2. **Equipment**

2.1. Which type of detector was used for the determination of Cs-137 in the air filter? *

- a) Ge(Li) detector
- b) HPGe detector
- c) BEGe detector
- d) Well type detector
- e) NaI(Tl) detector
- f) Other

2.1.1. If other, please specify here: *

2.1.2. Provide the supplier and the model of the detector and its relative efficiency:

2.1.3. Describe how the efficiency calibration of the detector is done and provide the source of nuclear data used:

2.2. What type of electronics and data acquisition was used? *

- a) Analog signal processing (spectroscopy amplifier, etc)
- b) Digital signal processing
- c) Other

2.2.1. If other, please specify here: *

Acquisition time and measurement cycles

Questions/Response table	*Acquisition time (sec)*	*Measurement cycles*	*Count rate for Cs-137 peak (counts/sec)*
Sample			
Background/blank			

Uncertainty budget

Questions/Response table	*Relative uncertainty component (%)*
Counting statistics	
Blank/background measurement	
Efficiency calibration (including the activity of the calibration source)	
Chemical yield	
Self-absorption in the source	
Activity of the calibration source	
Other 1	
Other 2	
Other 3	

2.3. Specify the software for peak area determination and data evaluation (more than one choice is possible): *

- ☐ a) Genie 2000 family
- ☐ b) GammaVision
- ☐ c) InterWinner
- ☐ d) Gamma-W
- ☐ e) EMCA+
- ☐ f) Gamma-track
- ☐ g) SAMPO
- ☐ h) HYPERMET
- ☐ i) Other

2.3.1. If other, please specify here:

3. Measurement and data evaluation

3.1. Fill in the following table: *
See table **Acquisition time and measurement cycles** at bottom

3.2. Provide information on the limit of detection (value in Bq, calculation method, etc.): *

4. Uncertainty budget
See table **Uncertainty budget** at bottom

4.1. If you indicated other, please specify the component(s):

5. Additional information

5.1. Difficulties encountered

5.2. Further comments on this interlaboratory comparison excercise:

Questions/Response table	*Relative uncertainty component (%)*
Combined relative standard uncertainty (quadratic sum of components)	

Annex 11: Responses to the questionnaire accompanying the reporting form

To determine the ratios, the 76 participants were taken as 100%.

Table A-11. Treatment of spiked air filters before measurement by the participants and equipment used for the measurement of the spiked air filters.

Question	Number of answers	Ratio (%)
"Were the comparison sample treated according to the same analytical procedure as routinely used in your laboratory for the same type of samples (^{137}Cs in air filters)?"		
Yes	60	79
No	16	21
"Did you measure the sample together with the plastic foil or did you measure the foil separately?"		
Together	29	38
Separately	27	36
The plastic foil was not measured	20	26
"Did you apply any preconcentration or chemical treatment?"		
Yes	0	0
No	76	100
"Which type of detector was used for the determination of ^{137}Cs in the air filter?"		
Ge(Li) detector	1	1
HPGe detector	60	79
BEGe detector	11	15
Well type detector	2	3
NaI(Tl) detector	1	1
Other	1	1
"What type of electronics and data acquisition was used?"		
a) Analog signal processing (spectroscopy amplifier, etc.)	24	32
b) Digital signal processing	52	68
c) Other	0	0

Table A-12. Measurement parameters.

	Minimum	**Maximum**	**Mean**	**Median**
Measurement time of sample (h)	1	168	55	48
Count rate for ^{137}Cs peak in sample (cps)	0.0004	0.2900	0.0156	0.0064
Measurement time of background (h)	0.08	434	86	69
Count rate for ^{137}Cs peak in background (cps)	0	0.0800	0.0025	0.0002
MDA (Bq)	0.000004	0.150	0.035	0.030

Annex 12: Uncertainty budget as reported by the participating laboratories.

Table A-13. Detailed uncertainty budget table reported by the participating laboratories.

Lab Nr	Counting stat.	Blank/ bkg meas.	Efficiency calc.	Chem. yield	Self-abs. in source	Act. of calib. source	Other 1	Other 2	Other 3	Comb. rel. std. unc.	Explanation of "other" components, notes
1										59.92	
2	10.2		1.2			3				10.4	
3	13.6		1			0.24				13.59	
4											
5											
6	23.56	0	3.37	0	0	1.5	5	0	0	24.32	Other 1: Systematic error
7	2.4	0	1.3	0	1.5	0.7	1			3.35	Geometry correction (counting efficiency correction for sample height).
8										12	
9	8.4	29.7	3							10.2	
10	14.9						5.3	2.5		16	other 1 = bkg + Efficiency calibration + Activity of the calibration source (k=2); other 2 = source weight + system stability (k=2)
11	7.95	0	7	0	0	0	0.24	0	0	8.92	Gamma photons emission probability
12	91.44	-	8.66	-	-	-	-	-	-		
13											
14	8.4	0	4	0	0	0	0.5	2		9.53	1-Self absortion in the sample; 2- radioactivity distribution on the filter
15	13.1		1.37				1	2		13.4	Other 1: additional systematic component; Other 2: additional random component

Lab Nr	Counting stat.	Blank/ bkg meas.	Efficiency calc.	Chem. yield	Self-abs. in source	Act. of calib. source	Other 1	Other 2	Other 3	Comb. rel. std. unc.	Explanation of "other" components, notes
16	3.5	0	6	0	0	0	Atenta-tion correc-tion	Homo-geneity	self emissi-on prob.	7.02	
17	4.3	8.02	6	0	0	0	1	0.24	2	11.1	Other 1-homogenity, Other 2-self emission probability, Other 3-atenuation correction
18	4.25	2	5				2			7.14	The uncertainty introduced by applying the efficiency transfer
19	7	0	6							9.219	
20	10.5		3.17			5				12.1	
21	1.6		5.1				1.4	2.2		6	Repeatability, Reproducibility
22	3.14	7.2	6	0	1	0	2	0.24	2	10.33	1. Sample homogeneity, 2. Cs-137 self emission probability, 3. Attenuation correction
23	2.8		3							4.1	
24	4.5	2.3	2.8							5.8	
25	5.5	-	3	-	-	3	2			6.5	Filter placement on the crystal
26										8.5	
27											
28	5.24	N/A	7.41	N/A	Zero		0.3	0.2		9.1	1 = Uncertainty of $T_{1/2}$; 2 = Uncertainty of gamma emission probability
29	10		4				2				Correction for density and fill height
30	4.4		6	0.24			5	4		9.82	Other source of uncertainty is the inhomogeneity and the lack of symmetry of the deposition of cesium on the sample

Lab Nr	Counting stat.	Blank/ bkg meas.	Efficiency calc.	Chem. yield	Self-abs. in source	Act. of calib. source	Other 1	Other 2	Other 3	Comb. rel. std. unc.	Explanation of "other" components, notes
31										24	combined uncertainty consists of experimental uncertainty, uncertainty in net peak area, uncertainty in calibration source, uncertainty in efficiency calibration
32	6		5			0.5				11	
33	6		1							5	
34	4.4	17.7	5	-	2	2				7.24	
35	6.9		3.5		0.65		0.264	0.235		7.8	Half-life and emission probability
36	100	100	100								Calculated by Genie 2K
37	2.33	3	4.5584			1.01				4.55	
38	5.257	Included in counting	3.98			included in eff.	0.24	0.8	0.5	6.67	(1) Branching Ratio, (2) Volume, (3) Geometry filling repeatability
39	3.8	-	0.9	-	-	-	1.4	0.5	-	4.2	Other 1: Difference between calibration source and measured sample (weight and size). Other 2: Uncertainty in positioning the sample on the detector.
40	3	3	3.6	0	0	3	2			6.63	geometry deviation between calibration source and pressed filter
41	5.3	5.3	-	-	-					9.8	
42	7.2		2				0.23				Other 1: Emission probability Cs-137 at 661 keV
43	3.72	3.92	1.00	-	-	1.20	-	-	-	<5%	
44	6	NA	5	NA	NA	NA	6			9.8	Other 1: sample preparation in plastic vessel
45	4.1		2.35			1.2	3	3.4		6.55	Relative uncertainty k=1; Other 1: flow measurement; Other 2: geometry uncertainty
46											

Lab Nr	Counting stat.	Blank/ bkg meas.	Efficiency calc.	Chem. yield	Self-abs. in source	Act. of calib. source	Other 1	Other 2	Other 3	Comb. rel. std. unc.	Explanation of "other" components, notes
47	2		3			1.5				3.54	
48			6								
49											
50	0.03	0.01	3.3	0	0.01	2.5				4.1	
51	6		5			5	10			14	coincidence correction. uncertainty used for every peak, as it is also used for the calibration curve.
52	5.7	0.5	4	na	0.01	2	2	2	6	22	Other 1 : summing correction; Other 2 : diameter; Other 3: density, composition, positioning, counting losses, system drift
53	5.4	2	3			0.7				6.5	
54	3.3	0	1.2	0.2	5	1.2	6			8.6	Uncertainty of sample preparation, homogeneity and geometry
55	9.26	-	1.45	-	-	0.81	-	-	-	-	
56										22	
57	2	13.5	2	0.15	0.1	1.4				14	
58	3.70		3.4 (44.8, 0.069)			(44.8, 0.069) Bq				4.90	
59											
60	1.26	in counting statstics	2.26	not applicable	not applicable	included in				2.6	
61	3.5	0	2	0	0	0	5			6.4	Correction for filter height (geometrical effect).
62	4	0	4	0	2	3				6	
63	37.5	35	3.5		22	2				12	
64	3	1	3		2					0.7	

Lab Nr	Counting stat.	Blank/ bkg meas.	Efficiency calc.	Chem. yield	Self-abs. in source	Act. of calib. source	Other 1	Other 2	Other 3	Comb. rel. std. unc.	Explanation of "other" components, notes
65	5	3	5	0	5	3	5	2		11	1. sample preparation 2. peak area determination
66											
67	0.99		5.99							6.07	
68	100										
69	4.2	0.0001	6							7.5	
70	3.28	0	6			0	0.235			6.84	Other 1: gamma yield uncertainty 0.235% (DDEP)
71	We do not, as of yet, report the individual contribution of different factors to the uncertainty budget. Instead we have a generously set value for random uncertainties, which includes factors such as sample weight uncertainty. This value is set at 5%. The uncertainties relating to the background, the counting statistics and the eff calib are dealt with in Apex but not displayed as separate posts										
72	1.6		6							6.6	The Combined relative standard uncertainty is not the quadratic sum of the components for a subtraction.
73											
74	1.4	30	0.043								
75											
76	1.61	0	2.14	0	0		0.27	0.02	6.7	7.2	(1): *u*(lambda); (2): *u*(gamma yield); (3): *u*(repeatability)

Table A-14. Comparison of uncertainties as reported by the participating laboratories and as calculated from the uncertainty budget table.

The second, third and fourth columns of this table contain the results reported by the participants in the reporting form. Relative standard uncertainty is calculated from the reported standard uncertainty and the reported activity. The last two columns contain the reported combined relative standard uncertainty as reported by the laboratories in the questionnaire and the quadratic sum calculated by JRC-IRMM from the given uncertainty components (calculated combined relative standard uncertainty), respectively (see *Table A-13*).

Lab Nr	Reported activity A (Bq)	Reported uncertainty $u_c(A)$ (Bq)	Reported k factor	Relative uncertainty $u_c(A)/A$ (%)	Reported comb. rel. standard unc. (%)	Calculated comb. rel. standard unc. (%)
1	0.0827	0.0495	2	59.85	59.92	
2	0.078812	0.016335	2	20.7265	20.8	21.4
3	0.0978	0.0142	1	14.52	13.59	13.64
4	0.066	0.005	1	7.6		
5	0.07	0.04	1	57		
6	0.077577	0.01887	2	24.324	24.32	24.37
7	0.085	0.006	2	7.1	6.70	6.69
8	0.0868	0.0098	1	11.29	12	
9	0.114	0.012	1	10.5	10.2	31.0
10	0.104	0.017	2	16.3	16	16
11	0.214	0.038	2	17.8	17.84	21.19
12	0.151	0.029	2	19.2		
13	0.11	0.008	2	7.3		
14	0.125	0.01	1	8	9.53	9.53
15	0.134	0.036	2	26.9	26.8	26.7
16	0.128	0.009	1	7.0	7.02	6.95
17	0.124	0.014	1	11.3	11.1	11.1
18	0.099	0.007	1	7.1	7.14	7.15
19	0.12247	0.02175	1	17.759	9.219	9.220
20	0.16	0.04	2	25	24.2	24.1
21	0.144	0.017	2	11.8	12	12
22	0.13	0.013	1	10.0	10.33	10.33

Lab Nr	Reported activity A (Bq)	Reported uncertainty $u_c(A)$ (Bq)	Reported k factor	Relative uncertainty $u_c(A)/A$ (%)	Reported comb. rel. standard unc. (%)	Calculated comb. rel. standard unc. (%)
23	0.168	0.014	2	8.3	8.2	8.2
24	0.161	0.009	1	5.6	5.8	5.8
25	0.153	0.01	1	7	6.5	7.2
26	0.18	0.015	1	8.3	8.5	
27	0.252	0.102	2	40.5		
28	0.206	0.0187	1	9.08	9.1	9.1
29	0.164	0.026	2	15.9		11.0
30	0.132	0.013	1	9.8	9.82	9.82
31	0.14	0.03	1	21	24	
32	0.184	0.021	2	11.4	11	8
33	0.21	0.01	2	5	5	6
34	2.05	0.25	2	12	14.48	38.24
35	0.19	0.03	2	16	15.6	15.5
36	0.184	0.041	2	22.3		
37	0.163	0.015	2	9.2	9.10	12.04
38	0.15	0.02	2	13	13.34	13.33
39	0.179	0.016	2	8.9	8.4	8.4
40	0.188	0.012	1	6.4	6.63	6.63
41	0.163	0.016	1	10	9.8	7.5
42	0.196	0.015	1	7.7		7.5
43	0.15	0.015	1	10.0	< 5%	6
44	0.212	0.02	1	9	9.8	9.8
45	0.245	0.026	1.65	10.6	10.81	10.99
46	0.235	0.026	1	11.1		
47	0.25	0.01	1	4	3.54	3.91
48	0.315	0.025	1	7.9		6.0
49	0.266	0.049	2	18.4		
50	0.302	0.012		4.0	4.1	4.1

Lab Nr	Reported activity A (Bq)	Reported uncertainty $u_c(A)$ (Bq)	Reported k factor	Relative uncertainty $u_c(A)/A$ (%)	Reported comb. rel. standard unc. (%)	Calculated comb. rel. standard unc. (%)
51	0.29	0.08	2	28	28	27
52	0.27	0.06	2	22	22	10
53	0.338	0.023	1.645	6.8	6.5	6.5
54	0.34	0.03	1	9	8.6	8.6
55	0.34	0.05	2	15	-	9
56	0.45	0.1	1.96	20	22	
57	0.5	0.07	1	14	14	14
58	0.318	0.015	1	4.7	4.90	5.02
59	0.47	0.05	2	11		
60	0.57	0.015	1	2.6	2.6	2.6
61	0.4723	0.0303	1	6.42	6.4	6.4
62	0.654	0.039	1	6	6	7
63	0.53	0.06	2	11	12	
64	0.6	0.06	2	10.0	10	10
65	0.65	0.07	2	11	11	11
66	0.565	0.029	2	5.1		
67	0.571	0.035	1	6.1	6.07	6.07
68	0.7	0.04	1	6		
69	0.493	0.037	1	7.5	7.5	7.3
70	0.803	0.0549	1	6.84	6.84	6.84
71	1.16	0.13	2	11		
72	1.09	0.07	1	6	6.6	6.2
73	0.099	0	0	0		
74	1.7424	0.11	2	6		30
75	1.46	0.07	1	5		
76	2.46	0.36	2	15	14.4	14.4

Annex 13: PomPlot

The PomPlot, an intuitive graphical method, is used to produce an overview of the results (Spasova et al., 2007). It displays the relative deviations (*D/MAD*) of the individual results *A* from the reference value A_0 on the horizontal axis and relative uncertainties (*u/MAD*) on the vertical axis (*Figure 8*). For both axes, the variables are expressed as multiples of *MAD*, which is defined as the median of absolute deviation from the reference value

$$MAD = Median|D_i|, (i = 1, \ldots, n) \quad (1)$$

where D_i is the difference between the reported and the reference activity concentration:

$$D_i = \frac{A_i}{A_0} - 1 \quad (2)$$

The median absolute deviation *MAD* is used because of its robustness.

For every datum point the uncertainty is calculated as an independent sum of the reported combined uncertainties on A_i and A_0

$$u_i^2 = u_c^2(A_i) + u_c^2(A_0) \quad (3)$$

where $u_c(A_i) = U(A)_i/k$ and $u_c(A_0) = U(A_0)/k$ (4)

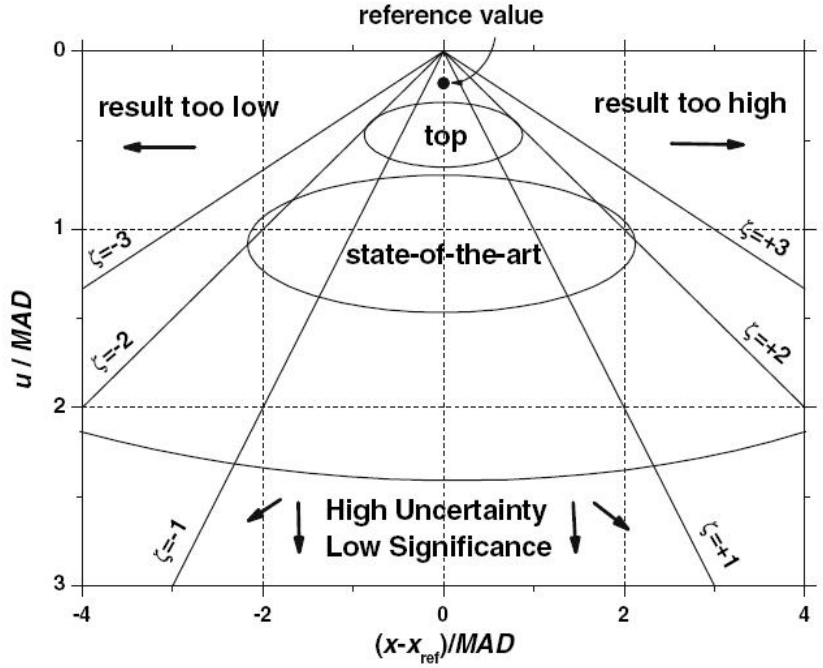


Fig. A-6. Interpretation of the PomPlot (Pommé, 2016)

The ζ-scores, |ζ|=|D/u|=1, 2 and 3, are represented by diagonal solid lines, creating the aspect of a pyramidal structure. The ζ-score is a measure for the deviation between laboratory result and reference value relative to the total uncertainty (ISO, 2005a). The points on the right-hand side of the graph correspond to results that are higher than the reference value whereas lower values are situated on the left. When the uncertainty is small, the corresponding point is situated high in the graph. The most accurate results should be situated close to the top of the pyramid. Points outside of the ζ=±3 lines are probably inconsistent with the reference value.

List of abbreviations and definitions

BIPM	Bureau International des Poids et Mesures
BEGe	Broad Energy Germanium detector
CIEMAT	Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas
CCRI(II)	Comité Consultatif des Rayonnements Ionisants, Section 2
DDEP	Decay Data Evaluation Program
EC	European Commission
EMRP	European Metrology Research Programme
EU MS	European Union Member States
EURATOM	European Atomic Energy Community
EURAMET	European Association of National Metrology Institutes
EURDEP	European Union Radiological Data Exchange Platform
GUM	Guide to the Expression of Uncertainty in Measurement
HPGe	High-Purity Germanium detector
ICS-REM	International Comparison Scheme for Radioactivity Environmental Monitoring
EC ILC	Interlaboratory comparison organised by JRC-IRMM
ISO	International Organization for Standardization
JRC	Directorate General Joint Research Centre
JRC-IRMM	JRC Institute for Reference Materials and Measurements
JRC-ITU	JRC Institute for Transuranium Elements
KCRV	Key Comparison Reference Value
LSC	liquid scintillation counter, liquid scintillation counting
MetroERM	Metrology for radiological early warning networks in Europe, ENV57
NIST	National Institute of Standards and Technology
NMI	National Metrology Institute
SIR	Système International de Référence, International Reference System for radionuclides
UTC	Coordinated Universal Time

A	activity measured by participating laboratory
A_0	activity reference value, spiked activity
$D_{\%}$	percentage difference
E_n	performance statistic E_n number
k	coverage factor according to GUM
MAD	median absolute deviation
MDA	Minimum Detectable Activity
stdev	standard deviation, standard uncertainty in counting alone

u_c	combined standard uncertainty according to GUM
U	expanded uncertainty according to GUM
$U(A)$	expanded uncertainty of laboratory result (k=2)
$U(A_0)$	expanded uncertainty of reference value (k=2)
$u(A)$	standard uncertainty of laboratory result (k=1)
$u(A_0)$	standard uncertainty of reference value (k=1)
$u_c(A/A_0)$	combined standard uncertainty of ratio A/A_0 (k=1)

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